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Investigation on Autonomous Healing for GFRP Composite using Cyanoacrylate Adhesives

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University of Dublin, Trinity College

A thesis submitted in fulfillment of the requirements for the degree of

*Doctor of Philosophy*

2012
Declaration

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Iyami owon, omo kehinde gbegbon
Ejire ara isokun, edunjobi omo edun tin sere ori igi
Wini wini loju orojun, ejiworo loju iya re
Okan ni mba bi mba yo, sugbon meji lowole tomiwa
Omo so ile alakisa di ile alaso
Thank you.
Summary

An investigation into smart structures with a vascular healing system using cyanoacrylate (CA) adhesive is presented in this thesis. The flexibility in the functional role of a smart structure involves the structure executing its primary operation(s) while also being at liberty to initiate damage repair without any external intervention. Many of the high performance engineering applications now utilise the light weight - high strength benefit of fibre reinforced polymer (FRP) composites as their main load bearing component. They are found in new generation sports vehicles, aircraft fuselage, megawatt wind turbine blades (WTB) etc. However, FRP composites are susceptible to the development of cracks from stress concentration, low velocity impact damage and are prone to delamination. Their failures are often characterised by insufficient warnings and can be catastrophic.

In this regard, a particular focus was placed on WTBs. Preliminary investigation was undertaken on a 6 kW glass fibre reinforced polypropylene WTB to examine the critical and the most failure susceptible sections along its length. The blade was instrumented with evenly spaced strain sensors along both sides of its aerodynamic shell and was tested statically and dynamically in flapwise movement. The static test result was validated using a finite element analysis.

The influence of out-of-plane stresses was examined through a mode I delamination failure on environmentally conditioned, CA bonded glass fibre reinforced plastic (GFRP) coupons. The coupons were initially subjected to DCB tests for their pristine fracture toughness and the delaminated halves were subsequently bonded back together using a CA adhesive. The coupons were then exposed to different simulated environmentally hostile conditions including different saline environment. The fracture toughness of each coupon was experimentally analysed upon withdrawal from their respective conditioning chambers of the
Henkel Loctite R & D facility in Ireland. It was observed that the CA bond strength was not compromised even under the influence of harsh environmental conditions.

The use of hollow glass fibres (HGF) embedded in the FRP structures, as self healing system, has been widely accepted as a way of combating microcrack development. However, HGFs critically undermine the effectiveness of self healing systems. They create opposing walls against propagating cracks and have been reported to be a potential impediment on the healing agent’s path. Therefore, a 2D hollow network channels within an FRP is proposed in this thesis. The efficiency of the vascular system with the use of a CA adhesive is investigated. A numerical tool was developed to aid the detection and modelling of the progressive manner of laminate failure.

The numerical model was aimed at the mechanical response(s) prognosis in FRP composites, through progressive failure in laminates. It uses logical material degrading model, Tsai-Hill failure criterion and a stated failure level to constantly examine each ply within a laminate. Accuracy of the prediction was compared with an experimental result and the conventional approach. It was found that the numerical model closely predicted similar experimental behaviour.

Furthermore, the vascular hollow channels of micrometer range diameter was designed and incorporated into a NACA 4415 WTB. The vascular network delivers the proposed multifunctional operation of a structure in such a way that mimics the haemostasis response in biological organisms. The network exhibits less impediments to sideways seepage on the transporting fluid. The flexural responses of the self healing structures were analysed through a three-point bending test at different stages. The test was carried out for damage initiation and for post-repair evaluation of the recovered efficiency.
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Chapter 1

Introduction

1.1 Motivation

The term 'composite' in a structural context refers to the combination of two or more materials, distinctively different in their properties, to form a new product with a homogeneous functionality, Figure 1.1. Common examples are: concrete's mortar/steel reinforcement, glass fibre reinforced plastic (GFRP) or carbon fibre reinforced plastic (CFRP), aramids fibre etc. Concrete represents a multi-composite material comprising of aggregates, mortar and reinforcing materials. In the context of this work, fibre strengthened composite is a mixed product with different materials comprising of reinforcements and binders. Generally, composites are of two different phases - the continuous and the discontinuous. The load carrying components, i.e the reinforcements are the fibres, are the discontinuous while the matrix represents the continuous phase, the weaker of the comprising materials.

Sustainable design requires innovative composite materials based on the structural and mechanical capabilities of existing materials such as the combination of light-weight reinforcing elements and polymer materials. Fuel efficient, light-weight and durable structures have been utilising fibre reinforced polymer (FRP) for decades (Richardson & Wisheart, 1996). In many cases, FRPs are adopted as the major load bearing component and as the industrial substitute for other
1. INTRODUCTION

materials such as metals. This is born out of the material's advantages of high stiffness to weight ratio, chemical inertness etc. FRPs are now used in industries such as aerospace, renewable energy, transportation and shipbuilding. The success of the material has propelled the use of CFRP and GFRP composites in advanced 21st century structures. For example Boeing's new generation of aircraft, the 787 Dreamliner, Figure 1.2, has the entire fuselage and greater portion of the wings made of CFRP materials. Also, GFRPs are now being used in longer wind turbines, Figure 1.3.

![Figure 1.2: Boeing 787 Dreamliner (Source: http://www.ledvista.ie/blog/)](http://www.ledvista.ie/blog/)

The major downside to FRP composites is their susceptibility to impact damage.
1.2 Wind Turbine blade

Figure 1.3: A typical long wind turbine blade - LM Glasfiber A/S: 61.5 m, 17.7 tons

FRPs are quite prone to sprawling cracks progressing to delamination. Superficial cracks or deep-seated damage of this nature can be difficult to detect visually. FRPs failures are often very sudden, preceded by little or insufficient warning to allow implementation of proactive measures. The inadequate alertness to FRPs imminent failure can be directly attributed to their operating nature. That is, under high stress, a crack can quickly transform to delamination and subsequent failure within a short period.

Therefore, a smart response system in FRPs is needed to detect and repair damage from the inception stage. This will in effect make the FRPs a multifunctional structure armed with an intrinsic healing functionality along with their primary mechanical roles. This operational flexibility of FRPs is currently being considered by many researchers (Dry, 1994; Kessler et al., 2003; Motuku et al., 1999; Nishiwaki et al., 2006; Pang & Bond, 2005b) for various structural applications such as aircraft, bridges etc. The self healing system discussed in this thesis investigates its application for wind turbine blades (WTB).

1.2 Wind Turbine blade

The blades are the central component of a wind turbine, generally made of FRP composites, and occasionally, made out of balsa wood for medium sized urban wind turbines. The turbines generate their mechanical power through the blades.
1. INTRODUCTION

The blades are often categorised according to the profile of their aerofoil cross-sections. The aerofoil shape enables efficiency in the aerodynamic functionality of the blades. The scale or size (length and width) of WTBs depends on desired power capacity of the turbine. Large scale wind turbines in recent times have been designed with longer blades, Figure 1.4.

![Figure 1.4: Increasing size of wind turbine (EWEA, 2010)](image)

1.2.1 Aerodynamics of WTBs

A synopsis of the aerodynamic aspect of a WTB is outlined. Further detailed information can be found in Hansen (2008) and Manwell et al. (2002). Wind turbine blades operate on the conventional principle of lift and drag. In a horizontal axis wind turbine (HAWT), Figure 2.9, the lift forces are caused by differences in the air pressure between the upper and the lower sides. A higher pressure difference on the lower side results in lift, a perpendicular movement of the aerofoil to the airflow direction. On the other hand, drag forces are caused by the combination
of viscous friction and unbalanced pressure on the surfaces leading to parallel movement in the wind direction. These forces play an important role in choosing material for blade design. respectively.

Figure 1.5: Typical wind turbine (Source: http://www.triplepundit.com)

Some important features of the blade's cross-section are illustrated in Figure 1.6. The *camber line* refers to the central line joining the points at halfway between the upper and the lower surfaces. The *leading* and *trailing edges* are the nearest and furtherest parts respectively, of the aerofoil into the wind. The *chord line* joins the two edges while the angle in between chord line and the relative wind is regarded as the *angle of attack*. The upper and lower surfaces are referred to as the *suction side* and *pressure side*

In larger wind turbine blades, 0.5 MW upwards, the aerodynamic shell contains
1. INTRODUCTION

a load carry box called spar. It runs through the length of the blades as shown in Figure 1.7. The spar has no aerodynamic function but provides structural rigidity and stiffness to the aerofoil shell. The spar supports majority of the load arising from flapwise bending of the blade through the flanges and shear web. The flanges stiffen and strengthen the blade against flapwise bending action from wind load, while the spar webs provide shear stiffness from shear loading. Flapwise bending refers to the bending of the blade on the rotating plane. Since the flanges are the main load carrying components, they usually contain composite laminates of 70 percent unidirectional reinforcing fibres for bending stiffness. The remainder consist of angly-ply layers for buckling resistance, especially on the low pressure side. The reinforcing fibres in the webs are typically in orthogonal direction, at ±45° for shear stiffness.

1.2.2 Increasing Size and Blade Materials

Over the past three decades, advancement in control methods and technology has seen installations of bigger wind turbines with longer blades covering over 120 m diameter of swept area, Figure 1.4. The increasing sizes are stirred by the possibility of increasing the output power from the turbines. Manufacturers tend to explore to the limits allowable by the guiding theoretical principles on the maximum power that can be generated based on equation 1.1

\[ P_{\text{wind}}(\text{watts}) = \frac{1}{2} \rho C_p A V^3 \]  

(1.1)
where $P_{\text{wind}}$ is the power available in the wind; $C_p$, power coefficient; $\rho$, air density; $V \text{ ms}^{-1}$, wind speed; and $A \text{ m}^2$, the swept area determined by the blade length. According to the equation 1.1, $A$ is a controllable team and is proportional to the amount of power, $P_{\text{wind}}$, that can be generated. However, blades cannot be allowed to be closer to the earth, hence the increasing tower height. Also, from equation 1.1, there is a “cubic effect” on the power harvested from wind, dictated by the wind speed, $V$. Wind speed generally increases further away from the earth surface, hence the increasing size of the wind turbines as shown in Figure 1.4.

As the tower height increases, the weight of the whole turbine becomes an issue, especially for the external rotating components. The weight of longer blades creates mass proportionality concern, as the blade mass is proportional to the cube of the turbine radius. As a result, large gravitational load is being generated on the turbine due to the large blades (Ashwill & Laird, 2007). Also, the blades are expected to be of sufficient stiffness to avoid them crashing into the turbine tower. These are the primary drivers for the use of FRPs in larger WTBs due to their high stiffness to weight ratio. Longer HAWT blades are now mainly made of
FRP, as it is the only suitable material at present, that is light weighted, of high strength and stiffness, and durable enough to function at high altitudes (Manwell et al., 2002), where there is higher wind load. At such heights, the total mass of the rotor blades and the nacelle posses a challenge.

In CFRP or GFRP WTBs, the reinforcing fibres account for 70% - 75% of a blade's weight. Depending on the designed structural capabilities, most of the fibres, in their matrices are oriented in the longitudinal direction parallel to the primary loading axis. Carbon/epoxy and glass/polyester polymer composites are the most generally used ones. But there are other various possible combinations (depending on design objectives) of these fibres with other matrices such as epoxy, polyester, and vinyl-ester. Glass fibres are the most commonly used reinforced fibres for WTB due to their cost advantage over carbon fibres.

1.3 Composites

Composite materials can be tailored to address specific structural needs. Hybrids (combinations of different types of matrices and, or different reinforcements) are often employed in fabrication of off-shore composite structures, collectively utilizing the properties of each component, the fibre and the matrix. Also, the tailoring strategy influences the form of the reinforcement used, in either fibrous or particulate composites. The latter is an isotropic composite reinforced with particles. The particles, uniform in shape and size, can be either randomly or dispersedly oriented within a matrix. Fibre reinforced composites are termed as fibrous composites. These consist of long or short fibres. The short fibres can be oriented as the particulate ones, while long ones are of single-layered composite (consisting of unidirectional or bi-directional lamina e.g 2D woven fabric) and multi-layered composite (configuration of different fibre orientation). Figure 1.8 illustrates the outlined classification.
1.3 Composites

Figure 1.8: Classification of FRP composites
1. INTRODUCTION

1.3.1 Reinforcement and Matrix

The most commonly used reinforcements in composite materials are glass fibre, carbon fibre, aramids (e.g. kevlar). They perform parallel functions to steel bars in concrete structures. Introduction to these fibres is limited to the main principal materials used in WTBs fabrication.

Glass fibre

Glass fibres consist of various oxide compounds such as calcium, sodium, iron, aluminium, boron and silicon. Silica ($SiO_2$), Figure 1.9, stands as the principal oxide constituting over 50% of glass fibres composition. These minerals are sourced from raw materials like sand, limestone and alumina which are then melted in a furnace at around 1260°C before being passed to a platinum bushing. The fibres flow out in filament form from several holes under gravity at the bushing base. A typical fibre diameter ranges from 8μm - 15μm.

Glass is amorphous but can be semi-crystalline at high temperature resulting in lower strength. The atomic structure of the fibres has major influence on the mechanical properties (strength and modulus). The silica molecules are arranged in a covalently bonded tetrahedral structure, sharing oxygen atoms between subsequent tetrahedral to create a rigid three-dimensional network (Hull & Clyne, 1996). This gives glass fibres an isotropic property of equal stiffness in both longitudinal and transverse directions.

Despite the high strength and economic advantage, glass fibres exhibit poor abrasion resistance, which reduces their serviceability strength. Their adhesion to some polymer matrix can be affected with moisture presence on their surfaces. At production, the fibre surfaces are chemically treated with sizing agent (e.g. starch oil) for abrasion resistance and coupling agent (e.g. silanes) to improve: adhesion, handling, reduce moisture effects etc. Glass fibres are manufactured commercially in different forms such as woven roving, chopped stand mat, fibre-glass fabric, and textile yarn. C-glass, E-glass and S-glass are the types of glass
fibre that are being produced. The E and S class are the most commonly used types in structural composites. E-glass is used in WTBs and other applications due to its low cost.

![Silica structure in crystalline form](image)

Figure 1.9: Silica structure in crystalline form

**Carbon fibre**

Carbon fibres (also known as graphite fibres) are of high modulus and strength, structurally stronger than glass fibres but relatively more expensive. Since 1990s, the price of carbon fibre has been reduced fuelling their use in high performance engineering applications. They are mostly used in aerospace industry and now in sporting goods, civil infrastructure, off-shore oil exploration etc. The fibres are manufactured from the pyrolysis of some organic materials such polyacrylonitrile (PAN) shown in Figure 1.10, pitch and rayon. In PAN manufacturing process, raw materials are converted to precursor fibre by whirling, followed by stretching, producing alignment of molecular chains. The stretched fibres are heated (between 205 - 240 °C) in an oxidising environment (air) producing ladder polymers.

Further heat treatment at approximately 1500 °C in an inert atmosphere removes non-carbon elements present in the fibres. This process is called carbonisation. Heat treatments after carbonisation and beyond 1800 °C regulate the percentage of carbon present in the fibre. The fibres with carbon content between 80% and
1. INTRODUCTION

The atoms in carbon fibres are arranged in sequential order, hexagonal in layers, similar in structure to graphite crystal. The atoms are densely packed in each sequenced layer, and held together by a strong covalent bond while weak Van der Waal forces bond the neighbouring atoms between different layers. This creates an anisotropic property unlike glass fibres. Therefore, high bond strength is exhibited along the densely packed layer plane resulting in high modulus in that direction and low modulus in the perpendicular plane. Thus, the transverse and shear properties of the fibres are influenced by the layer plane arrangement in the fibre's cross-section.

1.3.2 Matrix

The reinforcing fibres cannot be loaded directly due to their small cross-sectional area therefore, an intermediate material is essential. In this regard, the matrix accommodates the fibres, allows stress transfer from the loading surface to the reinforcing fibres, and also allows stress sharing between the fibres. Matrix plays the important role as a binding material. It also protects the reinforcing fibres from handling damage and environmental attack due to their chemical inertness. Their properties such as viscosity, reaction with fibres, melting and curing temperature influences the quality of the composite at the fabrication stage. Commonly used composite matrices are polymers, metals and ceramics. Polymer matrices are the most widely used matrices in commercial WTBs fabrication. Polymers exhibit light molecular weight, easy processing, and inertness to some common
environmental chemical substances. They are limited by their low melting point, low modulus, and degradation when exposed to some solvents and ultraviolet light. Polymers are either classified as thermosetting or thermoplastic depending on their respective physical and chemical properties.

Thermoplastic polymer consists of linear or branched-chain molecules and monomer units of high molecular weight. It exhibits strong forces (intramolecular bond) between the atoms and weak bonds between the molecules (intermolecular bond). Examples of this polymer include polyethylene, nylon, polycarbonate, polyether-ether ketone (PEEK) etc. These polymers soften or melt on heating. Under pressure or through thermal process the chemical structure (usually amorphous or semi crystalline) can be changed making possible reversible process of melting and solidification, and reshaping (Agarwal et al., 2006).

Thermoplastic polymers are highly viscous which raise workability issues. On the other hand, epoxy, polyester, vinylester etc. are common thermosetting polymers. They are found useful in WTBs manufacturing process. They do not melt but instead decompose on heating. Unlike the thermoplastics, thermosetting polymers consist of cross-link networks formed during polymerisation (curing). Beyond the curing stage, the material cannot be reshaped. The polymerisation process of most thermosetting resins is an exothermic reaction; external heating is optional for faster rate of reaction. In this reaction, liquid resins are converted to firm solid by cross-link formation, which creates covalently bonded molecules in a three-dimensional network.

Generally, the mechanical properties of polymer are affected by the constituent chemicals in the polymer, which dictates the network’s molecular units. Also, the quality of the curing process influences both the length and density of the cross-links formed as a product of the reaction. The following paragraphs elaborate on the common thermosetting polymers used in commercial WTBs fabrications.
Polyester

Polyester, with global production rate of two million tonnes per annum, is arguably the most widely used polymer in many composite applications. This is due to its economic advantage and easy processing. Polyester matrix is formed from precursor material, a prepolymer (polyester). It is an unsaturated resin dissolved in a large amount of polystyrene to form the final product.

The precursor, a lower molar mass material, and unsaturated liquid polyester, is formed from co-polycondensation of glycols (e.g. propylene glycol, diethylene glycol) with various anhydrides such as maleics or fumanics, and ortho or isophthalic anhydrides. The introduction of phtalic to the prepolymer lowers both the unsaturation content and the cross-link density. It also improves the behaviour at high temperature. Dissolving the precursor in polystyrene causes radical polymerisation based on the double carbon bond (covalent bond) in each reactant. The polymerisation process, an exothermic reaction, is initiated by small quantity of free-radical initiator or etherification catalyst such as benzoyl peroxide. There are no by products from the reaction but shrinkage sets in as a result of the high temperature involved.

The mechanical properties of polyester matrix can be modified by varying the nature or quantities of the constituting monomers in the precursor composition. Compared to epoxy, polyester is a brittle material due to the high proportion of polystyrene in its formulation. Partial or full replacement of the styrene compound could improve the polymer structure.

Epoxy

Epoxy resins are more expensive and lighter than their polyester counterpart. They are of; good adhesion, good resistance to chemical or hydrolysis, display good thermo-stability and less shrinkage during curing compared to polyester. It has an annual production of 0.8 million ton and it is used in electrical application
and structural components in aggressive environments. It is an organic liquid with the molecules in ring structured groups. Each group is made up of one oxygen and two carbon atoms.

Solid network of cross-linked epoxy polymers are formed from an exothermic reaction, similar to polyester’s production. In the formation, a precursor (liquid epoxy) is allowed to react with a hardener to form the matrix. The precursor is a linear polymer made from the reaction of epichlorohydrin and bisphenol A, called the diglycidyl ether of bisphenol A (DGEBA). The syntax A stands for synthesis from acetone. To produce an epoxy matrix via polymers cross-linking, the resins or precursors are made to react with an auxiliary agent, hardeners - usually amines, to polymerize the resins. The hardeners often dictate the level of curing temperature needed.

Like polyester, the properties such as molecular weights, are influenced by the properties of the constituting chemicals in the precursor and hardener. Also, differences in physical quantities e.g. by varying the proportion of the reactants affect the resin’s properties, which can be modified to obtain desired mechanical properties.

Vinyl ester

Vinyl esters are another thermosetting polymer used in structures like reaction vessels and pipes. The resin is produced by an esterification reaction of an epoxy resin and unsaturated monocarboxylic acid before the addition of styrene for thinning. The thinning process enhances the workability at the expense of strength depending on the additive’s proportion in the reaction. Vinyl ester is considered an hybrid of epoxy and polyester resins; the latter strengthened by the former. As the resins’ properties comparison shows in Table 1.1, the bulk properties of the vinyl ester are intermediate between epoxy and polyester resins.
1. INTRODUCTION

Epoxy resin structural formular made from Epichlorohydrin and Bisphenol-A

Figure 1.11: Chemical formula for an Epoxy resin

<table>
<thead>
<tr>
<th>Property</th>
<th>Epoxy</th>
<th>Vinyl ester</th>
<th>Polyester</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density, g/cm³</td>
<td>1.2-1.3</td>
<td>1.12-1.32</td>
<td>1.1-1.4</td>
</tr>
<tr>
<td>Tensile strength, MPa</td>
<td>55-130</td>
<td>73-81</td>
<td>34.5-103.5</td>
</tr>
<tr>
<td>Tensile modulus, GPa</td>
<td>2.75-4.10</td>
<td>3.0-3.5</td>
<td>2.0-4.4</td>
</tr>
<tr>
<td>Thermal expansion, 10⁻⁶/°C</td>
<td>45-65</td>
<td>53</td>
<td>55-100</td>
</tr>
<tr>
<td>Water absorption, % in 24 hours</td>
<td>0.08-0.15</td>
<td>–</td>
<td>0.15-0.6</td>
</tr>
</tbody>
</table>

Table 1.1: Common properties of cast epoxy, vinyl ester, and polyester resins (Agarwal et al., 2006)
1.4 Research Aims

Like the polyesters, they are low in viscosity, faster polymerization rate but more expensive. They are more chemically inert, even to wet environment; superior to polyester in physical properties, and handles better than either of polyester or epoxy resins.

The secret behind vinyl ester’s superiority lies in the molecular structure. When a polymer suffers from chemical attacks, it is usually a direct result of a breakdown through hydrolysis of the ester groups in the molecular structure. Unlike polyester matrix, there are few ester groups liable to hydrolysis and they are located outside the main body, Figure 1.12. Therefore, the main molecular chain is being shielded by the external esters, even if attacked they (main chain) will remain intact.

![Vinylester Resin](image)

Figure 1.12: Chemical structural formula for Vinylester resin

1.4 Research Aims

This thesis aims is to investigate the use of self healing technique in repair of crack damages in a WTB. The detrimental effects of crack growth and delamination on FRP composite structures have been widely published. Thus, an autonomous healing system is considered a pre-emptive measure to stop crack propagation or
1. INTRODUCTION

the progress to delamination. This thesis will investigate the use of hollow vascular network system as part of an FRP structural edifice for delivering a single part adhesive, cyanoacrylate, into a composite WTB. To achieve this, the goal is divided into smaller objectives:

Identification of damage prone site
A diagnostic experimental test will be conducted on a wind turbine blade for vulnerable sections along the length. The blade’s response to a realistic wind load which produces a flap-wise movement and static load will be examined. Also, finite element model of the test will be developed to validate the results obtained from the experimental phase.

Identification of damage prone FRP layer
In other to initiate an effective response system, progressive failure of FRP composite layers will be studied. And the formulation of a possible failure model of an FRP laminate, under flexural loading, will be developed as a damage predictive tool to single out the most susceptible layer(s).

Toughness of cyanoacrylate adhesive
The suitability of cyanoacrylate adhesive as potential healing chemical will be examined through series of double cantilever beam test, on FRP laminates bonded together and exposed to different environmental conditions.

Vascular network
An inherent, interconnected micro-channel network in an FRP laminate, will be designed to facilitate global coverage of the healing agent’s (cyanoacrylate adhesive) delivery, especially near any damage site. The knowledge from the above objectives will be used in transferring the self repair technique into a wind turbine blade made from fibre reinforced polymer composites.
1.5 Organisation of the Thesis

This thesis is divided into eight chapters following this chapter.

Chapter 2 presents the review of literature on FRP laminates, an outline on the elementary principles at a lamina level, their failures - on how it evolves from macro-cracks to delamination, and the available repair options with an extended view on bio-mimetic self healing approach.

The chemistry of potential healing agents are discussed in Chapter 3 with a particular focus on cyanoacrylate adhesives. It explains the adhesives' suitability for a self healing system, the necessary criteria for sufficient bonding to take place, and the characteristics of the class of the adhesive used for the entire test reported in the succeeding chapters.

Chapter 4 explains the preliminary experiments and finite element analysis on diagnostic approach directed towards finding the stress hotspots in a glass-polypropylene WTB.

Chapter 5 deals with the evaluation of fracture toughness of repaired delaminated GFRP coupons after they had been exposed to different simulated environmental conditions.

The design of a two dimensional hollow network at two different scales or sizes is implemented and the efficacy is experimentally tested in Chapter 6. It elaborates on how the test was conducted for differently proposed failure modes.

Chapter 7 presents a numerical method by which a self healing system can be optimised based on its proximity to the likelihood of failure location. It explains how different failure theories can be used with logical models to predict failure location within a laminated structure.

The penultimate chapter, Chapter 8, explains an experimental procedure into
1. INTRODUCTION

development of a dual functional structure. It describes how a small WTB with an intrinsic self healing function can be design, built and tested.

The concluding chapter summaries the results along with the closing remarks. It highlights how this thesis has contributed to the development of self healing composite. It also provides recommendation on how the work can be further enhanced and developed.

1.6 List of Experiments

A short summary of all the tests reported in this thesis are provided below:

- **Chapter 4** - Static and dynamic test on 6 kW WTB. Testing to show strain mapping along the length of the blade on both sides.

- **Chapter 5** - Determination of the fracture toughness of glass fibre reinforced polyester laminates to delamination, of different fibre types and under different environmental conditions. Woven glass fibres are used in the ambient weather condition, and the unidirectional glass fibres are used for different saline and cold temperature environments as well as for a control set under normal conditions. The ambient group contains six specimens labelled \( DB_1 \) to \( DB_6 \), while the environmental group has sixteen specimens divided into four groups of four specimens. The specimens from the latter group were labelled according to their environmental conditions.

- **Chapter 6** - Examines the flexural testing of two different sizes of GFRP composites. A bulky set was made of unidirectional fibres and contained drilled network of holes as delivery passages. The four specimens made were alphabetically labelled \( A \) to \( D \). The second flexural three-point bending test describes a test on slender woven GFRP laminates with inherent delivery network of micrometer range diameter. This test is further divided into two categories:
1.6 List of Experiments

- Fatigue test on specimens labelled $F1$, $F2$, and $F3$. Load is applied and taken off the specimens under three-point bending test for a stated number of cycles.

- A single three-point test is conducted on specimens with identification number $LD1$ to $LD5$. The test is terminated at an indication of a predefined level of strength degradation read from the load-deflection chart. Both tests are devised to initiate damages of small crack widths (micro cracks) and also to optimise the efficiency of the adopted healing chemical.

+ Chapter 8 - Examines the feasibility of creating a WTB with an intrinsic self healing capability, aided by in-built micrometer network channels for repairing fluid(s). A fabricated NACA 4415 WTB is simply supported and tested in flexure.
1. INTRODUCTION
Chapter 2

Literature Review

2.1 Introduction

This chapter provides an overview of published literature relating to the research topic and those discussed in the succeeding chapters. It begins with background theories and failures of FRP laminates. It discusses crack and delamination, their formation and growth as part of the associate failures. It finishes with the review of some potential self healing approaches for wind turbine blades; from design, manufacture/fabrication and operational perspective. It explains these methods—micro-encapsulation and vasculature of hollow fibres— their advantages and associated drawbacks.

2.2 Laminates

Lamina refers to the smallest scale of FRP composite, which consist of a layer (also known as ply) of structured reinforcing fibres cast in a matrix. The fibres’ structure from the manufacturer can be in the form of roving mat, woven roving mat, textile yarn, fabric etc. A stack collection of this lamina becomes a unit structure known as Laminate. Laminates are made through a subtle sequential process of laying fibre mats on a tacky matrix surface. The fibres can be oriented to suit a particular mechanical requirement in strength and stiffness. A lamina
or laminate can be oriented unidirectionally (all fibre in parallel direction), multidirectional (layers having different orientation angles) and likewise, random fibre orientations are often common.

Most particulate reinforced polymer composites are of the latter orientation and are considered isotropic - having equal properties in strength and stiffness in all direction. These two mechanical properties are generally controlled by the fibres angles, where the direction parallel to the fibre is the longitudinal direction and the direction perpendicular to the fibre is the transverse direction, Figure 2.1a. These directions are also the material’s axes. The longitudinal direction (1) is considered as the primary loading path therefore, a unidirectional FRP composite is stronger in the longitudinal direction than other directions (2 & 3) which have equal fibre distribution and hence similar strength.

Figure 2.1: a) Schematic representation of a unidirectional lamina, b) Developmental phases of composite structure, after Daniel & Ishai (2005)
2.3 Mechanical Properties of Laminates

Longitudinal Modulus

The longitudinal fibres, Figure 2.1a, oriented in the loading direction, primarily dictate the laminate's properties. Assume, a laminate composite illustrated in Figure 2.1a, to be of linear elastic behaviour with equal Poisson's ratios of the components in all direction (i.e. ignoring lateral contraction) having a fibre volume fraction of $V_f$, and matrix volume fraction of $V_m$ such that

$$V_f + V_m = 1 \quad (2.1)$$

Since no distinctive deformation exists between the fibre and matrix and hence leads to equal strain. The elastic modulus can be developed using this 'iso-strain' model where under-scripts, $c$, $f$, and $m$ stand for composite, fibre and matrix respectively. The strains are such that

$$\epsilon_c = \epsilon_f = \epsilon_m \quad (2.2)$$

The Young's modulus for the components are $E_f$ and $E_m$ for the fibres and matrix respectively. Similarly, their cross-sectional areas are $A_f$ and $A_m$. The uniaxial load $P_c$, on the laminate is split between the materials, such that

$$P_c = P_f + P_m$$

Given that

$$P_c = \sigma_c A_c$$

where $\sigma$ denotes stress and
2. LITERATURE REVIEW

\[ V_f = \frac{A_f}{A_c}; \quad V_m = \frac{A_m}{A_c} \]

We have

\[ \sigma_c A_c = \sigma_f A_f + \sigma_m A_m \]  \hspace{1cm} (2.3)

Dividing across equation 2.3 with the respective strain gives

\[ \frac{\sigma_c A_c}{\epsilon_c} = \frac{\sigma_f A_f}{\epsilon_f} + \frac{\sigma_m A_m}{\epsilon_m} \]

or

\[ E_c = E_f V_f + E_m (1 - V_f) \]  \hspace{1cm} (2.4)

as the longitudinal modulus of the laminate. This expression is commonly referred to as \textit{rules of mixtures} and can be written in a generalised form as

\[ E_c = \sum_{i=1}^{n} E_i V_i \]  \hspace{1cm} (2.5)

**Transverse Modulus**

Similarly, the transverse modulus of the laminate can be developed using the 'isostress' model, equal stress across the components, that is,

\[ \sigma_c = \sigma_f = \sigma_m \]

It is assumed that the cumulative thickness (identified by t) of either the fibre or the matrix across the cross-section reflects equal proportion of their respective volume fraction.
2.3 Mechanical Properties of Laminates

\[ V_f = \frac{t_f}{t_c}; \quad V_m = \frac{t_m}{t_c} \quad (2.6) \]

This implies that both matrix and fibre contributes to the composite elongation, \( \delta_c \).

\[ \delta_c = \delta_f + \delta_m \quad (2.7) \]

\[ \delta_c = \varepsilon_c t_c \quad (2.8) \]

\[ \varepsilon_c t_c = \varepsilon_f t_f + \varepsilon_m t_m \quad (2.9) \]

The strain is written in terms of the volume fraction in equation 2.10 by dividing both sides of equation 2.9 by \( t_c \) and assuming elastic deformation. The elastic modulus of the composite can then be expressed as shown in equation 2.11 in terms of the elastic modulus of the different constituting materials, using the stress-strain relation

\[ \sigma = \varepsilon E \]

and the 'iso-stress' model.

\[ \varepsilon_c = \varepsilon_f V_f + \varepsilon_m V_m \quad (2.10) \]

Or as

\[ \frac{1}{E_c} = \frac{V_f}{E_f} + \frac{V_m}{E_m} \quad (2.11) \]

The transverse modulus is commonly referred to as the inverse rule of mixture and can be written in a generalised form as
2. LITERATURE REVIEW

\[ E_c = \frac{1}{\sum_{i=1}^{n} \left( \frac{V_i}{E_i} \right)} \quad (2.12) \]

There are some ambiguities with the assumed models, particularly the 'iso-stress' model. Equal Poisson’s ratio assumption implies that existing constrains like strain concentration between the fibre and matrix are ignored. The ‘iso-stress’ principle assumed that the transverse and longitudinal stresses are equal which is generally true for isotropic materials and not for anisotropic materials. Rarely do the model predictions agree with experimental readings.

Shear Modulus

Henceforth the strength and modulus will be represented relative to the respective material axis, e.g longitudinal modulus \( E_c \) and transverse modulus \( E_c \) will be \( E_1 \) and \( E_2 \) respectively. The shear modulus, \( G_{12} \) (denoted by \( \tau \)) can be formulated using a similar model as the transverse modulus of equal shear stresses across the material plane, that is,

\[ \tau_c = \tau_f = \tau_m \quad (2.13) \]

Also the composite experiences a collective deformation such that

\[ \Delta_c = \Delta_f + \Delta_m \quad (2.14) \]

while the individual deformation is the product of the material’s shear strain, \( \gamma \), and thickness, \( t \). Consequently, deformation at each phase can be expressed as

\[ \Delta_c = \gamma_c t_c; \quad \Delta_f = \gamma_f t_f; \quad \Delta_m = \gamma_m t_m \quad (2.15) \]

This implies that \( \Delta_c \) can be written as
2.3 Mechanical Properties of Laminates

\[ \gamma_c t_c = \gamma_f t_f + \gamma_m t_m \]  \hspace{1cm} (2.16)

or in terms of volume fraction since

\[ V_n = \frac{t_n}{t_c}; \quad n = f, m \]  \hspace{1cm} (2.17)

\[ \gamma_c = \gamma_f V_f + \gamma_m V_m \]  \hspace{1cm} (2.18)

Under the assumption of linear shear stress and shear strain behaviour, corresponding shear stress and shear modulus relation, \( \frac{\tau_c}{G_m} \), can be substituted for each component’s shear strain, \( \gamma \), as in equation 2.19.

\[ \frac{\tau_c}{G_{12}} = \frac{\tau_f}{G_f} V_f + \frac{\tau_m}{G_m} V_m \]  \hspace{1cm} (2.19)

It can be further simplified to

\[ G_{12} = \frac{G_f G_m}{G_m V_f + G_f V_m} \]  \hspace{1cm} (2.20)

In a similar fashion the Poisson’s ratio, \( \nu \), can be deduced using the strain in longitudinal and transverse direction of a laminate with reference to the loaded side. For an isotropic material (with equal mechanical strength in all directions), the Poisson’s ratio is the negative ratio of the lateral strain, \( \varepsilon_2 \), to the longitudinal strain, \( \varepsilon_1 \), that is

\[ \nu = -\frac{\varepsilon_2}{\varepsilon_1} \]

However, for non-isotropic materials, when the loading or stress direction is along
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that of the majority of the fibres, the Poisson’s ratio, $\nu_{12}$ is considered major, and $\nu_{21}$, is considered minor and vice versa when it is otherwise (i.e when the stress is applied on side of plane 2, Figure 2.1a). Due to the high proportion of load in the longitudinal direction, $\nu_{12}$ is normally larger than $\nu_{21}$. In a similar fashion to the derivation of $E_1$, the $\nu_{12}$ can be predicted as

$$\nu_{12} = \nu_f V_f + \nu_m V_m (1 - V_f)$$  \hspace{1cm} (2.21)

and the minor Poisson’s ratio, $\nu_{21}$ can be represented as in equation 2.22 after further development of equation 2.21.

$$\frac{\nu_{21}}{E_2} = \frac{\nu_{12}}{E_1}$$  \hspace{1cm} (2.22)

2.3.1 Failure of FRP composite

The structural failures of WTBs and some FRP structures are seldom ever in less dramatic fashion. The failures are often accompanied by devastating penalties (Board, 2004). Most of FRP failures are characterised by a few obvious transient warnings. A failure can be an evolving process of crack initiation to delamination, and possibly buckling before failure. The more obscure the starting flaws are from the superficial level, the more difficult it is in devising a strategic preventive measure. A major initiator are microcracks buried beneath or on the surface. The studies in this thesis are centred on preventing such indistinct damages arising from cracks developed either from low velocity impactor on FRP composites or from internally generated stress. Such barely visible (impact) damages, (BVID) can result to delamination. BVIDs are usually small damages undetected under heavy maintenance and visual inspection under normal lighting condition from five feet.

Matrix Cracking

Microcrack formation arises from impact strike on FRP composite such as: tools dropping, birds & hail stone strikes, or from manufacturer defects such as local
stress concentration from polymer matrix curing to inappropriate manipulations of the reinforcing fibres. During the fabrication process, some polymer matrix (the thermosets) are polymerisation products of an irreversible exothermic reaction involving contraction, which generates internal stress within the matrix, (Hare, 1996). On the manufacturer’s part, design flaws or negligence in fabrication, generally referred to as “imperfections” give rise to susceptibility of internal stress leading to crack formation. It has been reported that non-operational induced defects such as void in matrix, misalignment or disoriented fibres, and unbalanced matrix concentration or volume are structurally detrimental to FRPs and can be a major contributor to their failures, (Marn et al., 2009).

A brief account of evolving failure from how BVID is presented chronologically from:

- Matrix cracking or crazing. This can be classified as intralaminar failure.

- Reinforcement fracture: occurs if the intensity of the impact is higher than capacity of the fibre, particularly for fibres oriented perpendicular or at large angles to the impact direction.

- Debonding between fibres and matrix develops in time. Fatigue sets in from cyclic loading and affected material begins to operate as a non-unique body. This is aided by the exposure of the fibres to environmental elements and possible accumulation of moisture.

- Delamination: Crack tip on the affected laminate acts as delamination onset between the crack-damaged laminate and the adjacent laminate. This is particularly true for cross-ply laminates (Agarwal et al., 2006). For a unidirectional reinforced plies, with crack in the transverse direction or at large angle to the load path, crack arrest is improbable at the laminate boundaries. Depending on impact force, the crack will propagate through the composite thickness until complete dissipation of the impact energy.
2.4 Delamination - Interlaminar Fracture

Delamination refers to the separation of two adjacent FRP layers in a laminate. It is also known as interlaminar fracture. It is an insidious failure. It is often located in the sub-inner layers and has generally been reported as FRP composite's major weakness (Bhushan & Stang, 2008; Maim et al., 2011; Mandell et al., 2003; Mikulik et al., 2008; Tay, 2003). Theoretically, delamination propagates from a crack onset if the strain energy release rate exceeds the laminate’s fracture toughness. It can arise from a number of sources: (i) internal generation of out of plane stresses, $\sigma_{xz}$ and $\sigma_{yz}$ (ii) fibre-matrix debonding (iii) the propagation of diagonally oriented cracks where they meet the interface between successive plies (iv) contaminated fibres (v) insufficient wetness of the reinforcing fibres by the matrix and (vi) imperfection from the fabrication process. The latter source can be caused by:

- **Voids**: Similar to the effect experienced in reinforced concrete, voids presence reduce the required or specified volume of the matrix; cause debonding; prevent proper adhesion and load transfer between the fibres and the matrix.

- **Resin**: Regions of different resin concentration create a non-homogeneous material. Non-isotropic properties are exhibited which consequently has a bearing on the mechanical properties.

- **Misalignment** of fibres placement or in their orientation can cause unexpected behaviour which can include flapping, twisting and buckling of the blades (Marn et al., 2009).

- **Sharp transition in shape profile** is structurally detrimental especially for FRP composite employed for aerodynamic applications. Re-entrant corner effect could be created, leading to high pressure of stagnant air within this region. In WTB profile, both in theory and practice, the hub is the most stressed part of the blade. This is the area of profile transition at wider angle, where the aerodynamic section and the cover are connected to the hub.
2.4 Delamination - Interlaminar Fracture

Interlaminar fracture is caused by accumulation of interlaminar stress which gradually reduces the matrix adhesion between adjacent layers in the out of plane direction — orthogonal direction to the transverse and longitudinal directions, Figure 2.1a. In FRP laminate fabrication or design, there is no inclusion of through-thickness reinforcement, as it is in reinforced concrete. The binding matrices, in between adjacent layers fill in this role in addition to their primary functions. Matrices are structurally the weaker component in a FRP composites and the presence of crack in them leads to gradually weaker structure over time. When this occurs, the homogeneity of such material is dislodged leading to rapid material disintegration.

2.4.1 Detecting Delamination

Far less work has been done in repair of delaminated composite structures. Much effort has been concentrated on the use of theoretical analysis and other various experimental methods—the non destructive technology of predicting and detecting delamination. Mandell et al. (2003), conducted a double cantilever beam test (DCB), destructive testing with a known degree of flaw and reported similar result of strain energy release from similar finite element analysis (FEA). Matsuzaki & Todoroki (2006) both worked on effective method of detecting delamination wirelessly based on changes in oscillating frequencies. Electrodes at opposite end of composite receive frequency signals, using the composite as the conducting medium. Delamination in the material causes increased resistance and varying frequencies at the receivers' ends. Similarly, Liu et al. (2012) attempted delamination detection using time-frequency representations (TFRs) through close identification of emitted Lamb waves produced as a result of the underlying imperfection in the structure. Analysis of discrete time-frequency signal gives the delamination location. Another detection technique involves the use of fibre Bragg grating (FBG) sensors (Grouve et al., 2008; Takeda et al., 2005, 2002; Todd et al., 2007). The FBGs consist of special functional filters — optical fibres. The fibres perform a selective transmission of wavelengths of lights in the filtering process. Some light rays are transmitted or allowed passage while the targeted ones are reflected. Takeda et al. (2002) observed sensitive changes in the FBG spectra as
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the delamination along the same plane of the embedded FGB sensor increases. Despite the susceptibility of the FBGs they are only localized, limited to small coverage unlike the lamb wave based approach (Liu et al., 2012).

Non-destructive Method of Detecting Delamination

Acoustic emission (AE): refers to the generation of transient elastic waves caused by sudden release of energy within a material. The subjection of a subsurface flawed material to external actions such as loading causes rapid stress redistribution (Agarwal et al., 2006). The stress is relieved in the form of sounds, sometimes audible to unaided ear. The released waves are picked-up by sensors. In composite, fibre fracture, matrix cracking, fibre-matrix interface damage and delamination are all characterized with high frequency transient acoustic signals. Recorded parameters such as peak amplitude frequencies of event times and duration, contained in the received signals hold vital information in analysing, identifying and locating the flaws. AE is different from other non-destructive techniques. It has no energy or wave input into the specimen but simply collects sound or stress waves from the specimen. It is more suitable to dynamic process such as crack and delamination propagation.

Ultrasonic Testing (UT): Involves the use of high frequency sound waves to evaluate the degree of flaws present in a material. It has been found to be quite useful for materials with large surface area, (Jorgensen et al., 2004). It is equally useful in taking measurements (dimensions), material characterisation etc. Typical UT system consists of a transducer, receiver/pulse or pitch, and a display device such as oscilloscope. High frequency energy generated by the transducer propagates through the test specimen in the form of waves. Discontinuity (such as delamination, voids, cracks etc) along the wave path causes reflected waves to be transmitted to the receiver via the transducer. Waves are transmitted normal to the specimen and some are reflected along same path in a method known as pulse-echo method.
2.4 Delamination - Interlaminar Fracture

The pitch-catch method involves transmitting the high frequency waves at other angles (Agarwal et al., 2006). These often require a second transducer as the receiver. The attenuated signal from the receiver is analysed electronically and displayed. Time varying amplitude of the pulses is displayed as the A-scan. A number of these signal at different point along a line gives a B-scan, while in two dimensional its creates a C-scan.

X-Radiography: Works on general principle of x-ray. Homogeneous materials without defects should possess equal density at every point, and hence equal absorption of x-ray beams. The photographic film of a material exposed to x-ray beams can be analysed for the presence of defects such as delamination, voids etc. Defect in materials causes reduction in their absorption coefficient. Thus, damaged sites can be located by cross-checking absorption coefficient against the expected result from a healthy material.

Thermograph: uses heat in diagnosing defects in materials. The resistance to conductance of a material depends on its flawlessness. Active and passive heating are the two methods used in thermographic testing technique. The latter involves heating specimens surface at high temperature using high voltage flash lamps for a short period. The surface temperature is monitored using an infrared imaging system. The warmer region would indicate presence of subsurface defects. Alternatively, the whole specimen can be preheated to homogeneous temperature; the coolest region will indicate the presence of subsurface defects. Passive heating uses internal heat generated from frictional effect on the defected surfaces under mechanical action such as cyclic loading. The warmest region indicates subsurface defects. The former method is thought to be more suitable to fatigue testing. Thermographic method is not as accurate as the other methods outlined but it is cheaper and faster.
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2.4.2 Preventing Delamination

In the aerospace industry it is thought that delamination can be prevented or delayed through the use of tougher resins and through other conservative methods in design and fabrication (Department of defense, 2002), thereby incurring more cost at the detriment of innovative approach. A number of approaches have been reported to address the issue of delamination. These include the use of bonding pins through the delaminated area (Li et al., 2006). Such repair techniques will be costly for components of large scale structures such as WTBs which have accessibility issues associated with the nacelle height and, in the case of off-shore WTBs, installation in remote locations adds significantly to maintenance costs (Larsen, 2009).

An alternative technique uses flax yarn as through-thickness reinforcement at the fabrication stage (Ghasemnejad et al., 2012). FRP specimens reinforced with flax z-pinned before matrix curing were shown to perform better in Charpy impact tests when compared with those without out-of-plane fortifications. A technique that has potential for use in remote locations is the use of piezoelectric patches (Alaimo et al., 2009; Wang & Quek, 2004; Wang et al., 2002). The electro-mechanical properties of the patches yield a range of uses in structural health monitoring (STM) where they can function as piezoelectric sensors or actuators. Their ability to produce electrical charge when mechanical pressure is applied, and vice versa, can be used in structural repair applications. The repair is apparent in the sense that the voltage supplied to the patch at the damage site causes the piezoelectric patch to change shape or deform which compensates for the induced stress or moment in the damaged structure (Wu & Wang, 2010). Such a repairing system implies a continuous power requirement to maintain the integrity of the structure.
2.4 Delamination - Interlaminar Fracture

2.4.3 Characterising Delamination

Fracture Toughness

Interlaminar fracture toughness, $G_c$, refers to the resistance of a material to delamination. Different methods are used in quantifying the resistive force or strain energy - a destructive method of testing. It is widely accepted that there are three modes of delamination. The splitting or opening mode I, sideways mode II and mix mode, Figure 2.2. The first mode is commonly found in WTBs in areas such as the spar’s webs and flanges, and in the edges.

![Figure 2.2: Delamination Modes](image)

The adjacent bond strength between two consecutive layers can be determined for mode I and mode II, $G_{IC}$ and $G_{IIc}$ respectively through some experimental techniques. There are available and widely used international standards by credible organisation such as the British Standard (BS), American Society for Testing Materials (ASTM), and Japanese Industrials Standard (JIS) for both modes on the quantification of the fracture toughness. Various methods have been suggested; mode I - double cantilever beam test (DCB), and tapered double cantilever beam test (TDCB); mode II - end notch flexure (ENF), end loaded split (ELS), four-point bend end-notched flexure (4ENF) and tapered end notch flexure (TENF).

Mode I - DCB

Double cantilever beam testing method, Figure 2.3, has been around since the 1980s. It is now a widely used tool (ASTM D5528-01, and BS ISO 15024:2001)
to quantify the mode-I interlaminar toughness of FRP composite. The test is carried out by equally loading both edges of a material (with initial crack length $a_0$) by force $P$ in opposite directions. This leads to tearing in the plane with least resistance i.e the plane with an intermediary material for crack initiator. Cracks propagate at a critical load resulting in decreasing $P$. The fracture toughness or energy, $G_{jc}$, is determined through data reduction process using the accumulated data — time, deflection, and corresponding crack length. The $G_{jc}$ cannot be considered as laminate material property but rather as the bond toughness at the fibre-matrix interface. Similarly, in the late 80s the European structure integrity society (ESIS) found out that thicker specimens showed increased toughness (Moore et al., 2001). Several mathematical methods are used in the data reduction process. These are outlined in Appendix A.

![Double cantilever beam set up](image)

Figure 2.3: Double cantilever beam set up

Mode II - ENF

The end notch flexure test, Figure 2.4 is the most widely use standard (ASTM WK22949) since the mid 1980s to examine a composite coupon resistance to
2.4 Delamination - Interlaminar Fracture

sliding or shearing mode of delamination. The same methodology of specimen preparation is shared with DCB except that the loads are applied in three point bending. This loading condition results in pure shearing at the crack tip (Johnson & Mangalgiri, 1985). The non-opening of crack under bending separates the two modes. Crack propagation depends on the specimen formation. Simple beam theory can be used in analysing the collected data (load, time and deflection) according to the equation 2.23:

\[ G_{IIc} = \frac{9a^2P^2}{16E_{1f}b^2h^3} \]  

(2.23)

where \( G_{IIc} \) is mode-II delamination fracture toughness, and \( E_{1f} \) is the longitudinal flexural modulus of the material.

More controversies have engulfed the acceptable method of testing the mode II interlaminar fracture. There are drawbacks associated with ENF as specimens can develop unstable crack patterns and longitudinal sliding. This has led to the introduction of the stabilised end notch flexure (SENF) and ELS. SENF (Davies et al., 1996) proposed an opening crack displacement, and ELS could not rectify the limitations either. Rather they were compounded with larger displacement.
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Over a decade after the inception, there has been no consensus on the preferable mode II method. The ASTM sides with ENF and JIS group preferred SENF.

2.5 Self Healing

The susceptibility to damage of FRP materials has been explicitly outlined in the preceding sections. WTB failures account for the highest reported incidents on wind turbines, including occasions of which blade has been reported to have flown hundreds of meters from the damage site (Caithness, 2011). Frequent failure of this energy generator undermines confidence in its sustainability not considering the planet’s sustainability at large. A non-operational wind turbine carries a serious financial consequence in blade replacement and fitting, possible power outage, loss of earnings etc. The biggest issue is averting sudden failure. FRP failures are unlike metallic based structure whose impending failure comes with noticeable warning. Failures of FRP composite progress through a failure developmental phase of most likely microcrack development to delamination or buckling. This eventual failure does not necessarily follow this chronological path always. Like a snowballing effect, failure originates from minor defects such as microcracks on the surface or on deep-seated layer leading to debonding of the fibre-matrix interface. Since the failure/delamination triggering effects can be invisible to the naked eyes, it is paramount that these instigating defects are combated before aggravation of their effects commence.

Self healing is one of the most actively researched auto-maintenance mechanism with the intended purpose of preventing sudden failures in structures. Self healing involves active repair of a component using materials already contained within. It involves design of smart materials for engineering applications with the benefit of optimized maintenance strategies. Self healing structures will become sensitive to some classified damages, and initiate instantaneous repair without external intervention, mainly human.
In terms of basic principle, it is an inherent healing function of a structure using on board curable agent (chemical(s)), released upon damage detection. It is analogous to biological activity in living organisms, therefore, mimicking nature. An example is blood clotting; the body responds to wounds and bruises through haemostasis system of blood coagulation. When bleeding occurs from damaged blood vessel linings, blood clotting is immediately activated. This natural healing process involves a series of complex biochemical mechanisms of platelets transportation to the damage site. It has been extensively investigated that wounds undergo some stages before healing can be generally accepted (Braiman-Wiksman et al., 2007; Tsirogianni et al., 2006). Upon wound detection, inflammation response kick-starts blood clotting. This is followed by proliferation of the cells involving transportation of dermal and the epidermal cells and matrix synthesis to cover the cut surface(s). Along the progressive healing stages is the skin tissue and skin restoration through tissue remodelling. Distinctively, plants (particularly trees) seal rather than heal damaged spots through compartmentalisation. It is a defence mechanism by which plants prevent the spread of infection from affected site to healthy parts. The healthy tissues seal off damaged areas that might be infectious to other healthy parts, whilst the wounded part is isolated from normal functioning and nutrient transportation is rerouted. Similarly, the synthetic healing procedure mimics the haemostasis response as shown schematically in Figure 2.5.

As an actively researched topic, self healing and other logically synonymous techniques have been considered for various engineering applications. Early pioneer of self healing concrete, Edvardsen (1999), reported that for a given crack width, increasing water pressure via crack plane would favour auto-precipitation of calcium carbonate crystal ($\text{CaCO}_3$) or calcite for steady healing. Calcite has been known for years to be the binder in sandstones and shales. More recently, similar effect was created when wet and drying cycles were introduced to certain cementitous concrete (Yang et al., 2009).

Another approach which has gained much relevance in the last decade with en-
Figure 2.5: Schematic of self healing procedure
2.5 Self Healing

couraging results is bacterial concrete, (Afifudin et al., 2011; Bang et al., 2001; Jonkers et al., 2010; Ramakrishnan et al., 2005). It is brought about by means of incorporating soil organisms which are capable of producing calcite to enhance damaged repair. The cementitious material, CaCO$_3$, is precipitated under favourable conditions through microbiological induction of soil bacteria such as Bacillus Pasteurii, Cohnii, Pseudofirmus, and Halodurans. With good measure of success, other innovative approaches to self healing concrete methodology include the use of shape memory alloys (SMA), independently or in conjunction with encapsulated healing materials (Kuang & Ou, 2008); and selective heating (SH) (Nishiwaki et al., 2006). The former system utilizes the unique advantage of SMAs to return to their original positions (post-deformation reaction) after which, if present, the on-board cementitious material can be released to the crack surface.

SH is somewhat different. It involves two overlying embedded pipes of different functionalities. One is a heat sensitive organic film pipe housing an embedded repairing agent. The other is the diagnostic component consisting of fibre reinforced composites and electro-conductive materials acting as a heating device. Electrical resistance in the thermal device changes with respect to strains developed in the concrete from crack growth. Partial increase in strain raises the electrical resistance, a consequence of the electrical conductive path been altered by the cracks. This results into localised heating around the crack area only, supplying sufficient heat to melt the organic film pipe thereby releasing the stored repairing agent onto the crack.

Following up on this intrinsic healing functionality of the present modern age materials, there are currently three actively researched ventures or methodologies particularly for FRP composites. Their technicalities are outlined below.

2.5.1 Micro-encapsulation

Micro-encapsulation has been constantly used in the food and agricultural industries, cosmetic & fashion industries, and medicinal science (Nack, 1970). However, employing it as a storage container adds a new twist to its growing utility scale.
and it is one of the most widely coveted self healing research approach. Encapsulation of autonomous healing agents in polymer composites has been actively considered for the last decade (Bleay et al., 2001; Kessler & White, 2001, 2002; Motuku et al., 1999; White et al., 2001). The technique involves having flimsy pockets of the repairing fluid widely dispersed in a polymer matrix for correcting or alleviating the adverse impact of specific defects arising from a structure’s operations. Encapsulating materials or vessels are generally designed to be more brittle in comparison with the polymer matrix. In spite of the brittleness necessary for crack penetration, they also form a protective and inert shield for either the healing agent or the catalyst. The inertness preserves the chemical properties of the on-board agent. Simultaneously, the shell prevents untimely reaction (of the enclosed fluid) with the neighbouring polymer matrix or changes in the fluid’s chemical properties due to environmentally induced conditions. Under this approach, an elaborated design of the microcapsule is fundamental to an efficiently active self healing application. Detailed microcapsule manufacturing processes are beyond the scope of this project. However, on a snippet view, in situ, interfacial polymerisation and meltable-dispersion are common with self healing capsule formation (Alexandridou, 2001; Cosco et al., 2007; Liu et al., 2009).

Successful use of evenly dispersed dicyclopentadiene (DCPD) monomer, within a polymer matrix as repairing agent, stored in urea-formaldehyde microcapsules, has been established (Kessler & White, 2001; White et al., 2001). As shown in the illustrative Figure 2.6, progressing crack ruptured the capsule in its path, thereby releasing the chemical content via capillary action into the crack(s) for contact-based polymerisation. The curing (preceding healing) process involves ring-opening metathesis polymerisation (ROMP) of evenly dispersed embedded catalyst (Grubb’s catalyst) and DCPD. The liquid healing agent, DPCD has been extensively used in micro-encapsulation based self healing application due to its availability, low cost, easy polymerisation when in contact with suitable catalyst, low viscosity, and most importantly endurance (Murphy & Wudl, 2010). Meanwhile the first generation of the Grubb’s catalyst, Figure 2.7 has been revealed to have some limitations such as deactivation. This has been attributed to per-
petual air or moisture exposure and reaction with some matrix resins such as diethylenetriamine (DETA), an epoxy curing agent (Jones et al., 2006). Also, agglomeration of its particles are undesirable (Kessler et al., 2003). However, soaking the particles in paraffin, prior to dispersing them into the hostile matrix, would retain their reactivity despite minute loss of activeness (Rule et al., 2005; Taber & Frankowski, 2003). Encouraging mechanical recovery efficiencies above 70% have been reported for self healing system through micro-encapsulation, (Kessler & White, 2001; Kessler et al., 2003).

![Micro-encapsulation three stages of healing](image)

Figure 2.6: Micro-encapsulation three stages of healing. a, formation of crack; b, release of healing agent from ruptured microcapsule; c, crack sealed by polymerization resulting from healing agent contact with catalyst, after White et al. (2001)

Both the proximity of the Grubbs’ catalysts to the microcapsule, and the fragility of the microcapsule’s walls are vital for any appreciable healing or strength recovery to take place. Accompanied with this are other challenges; voids created by emptied capsule can be damaging to the mechanical properties of the incorporating material. The voids may act as prospective sites for delamination initiation.
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Other disadvantages include fibre architecture disruption, probable unevenly dispersion and limited volume of repairing agent.

2.5.2 Vascular System

Vascular network of hollow tubes incorporated into a composite structure presents a different proposition to addressing small but potential damaging flaw at the infancy stage in a most labour and cost efficient way. Work on vascular system has evolved over time, starting from embodiment of one dimensional (1D) (Bleay et al., 2001; Dry, 1994; Motuku et al., 1999; Pang & Bond, 2005a,b; Trask & Bond, 2006) hollow glass fibres (HGF) in between vulnerable layers of FRP composite (Williams et al., 2007) to (2D) (Williams et al., 2008a,b,c), and now (3D) HGFs (Hansen et al., 2009; Toohey et al., 2007, 2009). There are no significant differences between this approach and that of microencapsulation. They both have similar design cycle and same repair activation mechanism namely, crack detection. The fragility of the HGFs allows penetrating cracks to create percolating openings for healing agent to be dispersed into the cracks. However, disparity exists in the fabrication and incorporation of both microcapsule and HGFs into the composite.

Micro-encapsulation system appears monotonic in objective with potential de-
lamination as adverse effect of emptied pods. In contrast, HGFs or the network version are multifunctional. They serve as additional reinforcement for the matrix (Bleay et al., 2001) without any appreciable structural detriment. HGFs enhance increased volumetric capacity of on board healing agents, likewise the external storage facility or reservoir for vascular system of HGFs. The extraneous chemicals can be replenished and manipulated to suit a desirable effect e.g adjusting the operating temperature, pressure and viscosity. Aside the primary healing function, HGFs can be strategically placed within the stacking sequence of the host material or oriented with the fibres to address anticipated failures.

2.5 Self Healing

One Dimension, 1D HGFs

One dimensional hollow passage, Figure 2.8, for healing agent had been an interesting subject of study since the early 1990s. Polymer hollow fibres or metals of same kind are deemed unsuitable due to the necessity of a controlled fracture upon impact (Motuku et al., 1999). Dry (1996) had conducted a conceptual and qualitative investigation on the feasibility of this repair mechanism on concrete using single part (cyanoacrylate), and two part epoxy adhesives as healing agents. Motuku et al. (1999) followed suit without any radical change(s) to the initial perceptive but employed a polymer composite of vinyl ester and epoxy resin as the repairable host. Their work focused on some logistic parameters, which are essential components of optimising the system, and therefore the recovered mechanical efficiency was not evaluated. Commercial HGFs commonly used in HGF self healing experimentation, are within the range of 15μm to 60μm diameter. The smaller sizes are preferable in order to keep initial mechanical properties, although filling them presents a different challenge. Bleay et al. (2001) developed a vacuum assisted filling technique, which had been adopted by many researchers. The authors had attempted to enhance the healing agent’s fluidity for improved viscosity by diluting it with some solvents. Pang & Bond (2005b) showed that this approach mitigates the effectiveness of the system due to undesirable chemical reaction between the solvent and the healing agent. However, they used a larger diameter HGFs for their test for effective filling capability. As reported by Hucker et al. (2003), larger HGFs diameter performs better under compression. Before
repair can be activated, damage(s) inconspicuous at times must be detected. This has made common the use of UV fluorescence dyes (Bleay et al., 2001; Motuku et al., 1999; Pang & Bond, 2005a,b) which are dispersed into the crack surfaces via the hollow fibres to aid visual inspection of damages and recovery.

![Diagram of single directional HGF concept](image)

**Figure 2.8**: A schematic illustration of the single directional HGF concept, after Bleay et al. (2001)

**Two and Three Dimension, 2D & 3D HGFs**

Higher dimension interconnected HGFs offer efficiency and optimal use of the self repair system, along with an extensive coverage. This is suitable for instantaneous repair of many types of composite failure originating from crack formation. It offers the prospect of replenishing repair agent(s) throughout the design life of such self healing structure. Similar ideology (Chang et al., 2009; Chung, 2004) is used in structures with thermoregulation applications, such as self heating, underground heating on bridges and airport runways. Though the higher order vascular system of 2D, Figure 2.9 and 3D are currently nascent but Williams et al. (2008a) had shown the feasibility of the system in an edge-wise compression test of a sandwich composite panel though no comparison was drawn against the
2.5 Self Healing

singular dimensional hollow fibre healing system.

Figure 2.9: 2D Network design for self-healing sandwich specimens. (A) Channel designed for resin component and (B) channel designed for hardener component, after Williams et al. (2008a)

Following the demonstration of the fundamental principles of 2D HGFs, efforts are now being shifted to variable parameters influencing the optimization of the adhesive flow in such a network. Williams et al. (2008c) further developed a paradigm for an optimised flow within a flow network as a function of the channel diameters modelled on Murray's law (Murray, 1926) of fluid flow in biological system. Murray reasoned that the power loss occurs in blood flow is caused by friction between the vessel walls; and went on to investigate the minimum power required to pump the fluid within a constrained volume to maintain constant flow rate. Smaller diameter vessels will require more pumping power to overcome friction. For minimum power, sum of the cubes of the daughter or branch vessels radii must equal to the cube radius of the parent vessel, equation 2.25. This is generally known as Murray’s law. Therefore, condition for minimum energy dissipated exists when the volumetric flow \( Q \), is proportional to the cube radius, \( R \), equation 2.24.

\[
Q \propto R^3
\]  \hspace{1cm} (2.24)
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\[ R_p^3 = R_{d1}^3 + R_{d2}^3 + \ldots + R_{dn}^3 \]  \hspace{1cm} (2.25)

where \( R_p \) is the parent vessel's radius, \( R_{d1}, \ldots, R_{dn} \) is the radius at each corresponding generation or branch.

The analysis presents a useful tool to optimize vessels' diameters for high performance engineering application considering that the network of these vessels will disrupt the material architecture. It is either the section size of the host material is increased to compensate for the volume displaced by the HGFs, or the cross-sectional dimensions of the vessels are reduced, thus increasing power input/needed for pumping. Hence, the need for a trade off between the power required and mass penalty (Williams et al., 2008c). Lim D (2003) examined the flow resistant in a four-generation non-cylindrical vascular network using a fluorescent dye for visual aid. The author reported samples with Murray's networks experienced 33% less resistant compared to the other samples which were not subjected to the theory—those kept as controls. This implies adopting bigger and constant cross-sectional dimension per generation will mean more bulky structure to maintain global stiffness.

However, 3D network of vascular system stands as the pinnacle of optimizing the system of self healing with extensive and effective coverage, Figure 2.10. The development is still at an infancy stage. Among the few contributed works to this field is the optimization modelling of a multi-purpose 3D flow network using genetic algorithm, considering objective and constrain parameters such as less resistive flow and volume (voids, channel diameter etc) respectively (Aragn et al., 2008). Toohey et al. (2007, 2009) developed a bulk epoxy matrix composite featuring a 3D vascular analogous to a functioning epidermic tissue. The hollow network was carved into the specimen using direct – inkwriting. The authors engaged the combination of the encapsulation technique and vascular system. DCPD monomer flows under capillary action from a fractured hollow enclosure and polymerises when it comes in contact with embedded Grubbs catalyst in the
2.5 Self Healing

matrix. The method also enabled multiple healing cycles.

Figure 2.10: A schematic 3D network for vascular system

The biggest challenge facing this second generation is the incorporation of the 3D network into a composite laminate. Higher dimension vascular system of self healing requires special fabrication methods such as direct ink writing. The system uses an omnidirectional printing technique whereby an ephemeral organic ink scaffold forms the basic structure of the vascular network. Thereafter, it is infiltrated with the intended polymer matrix. The scaffold is then removed through a liquefaction process. Another method with an exciting prospect for higher order hollow connectivity involves rapidly releasing or discharging accumulated electrostatic chargers from an electron beam irradiation. It has been experimentally demonstrated on solid polymer, polymethyl-methacrylate (PMMA) and polylactic acid (PLA), (Huang et al., 2009). The electron discharging method involves irradiating a substrate, in this case — the vascular host, with high level electron beam. After a sufficient accumulation of the electrons, the material is discharged with such intensity that the neighbouring material experiences fractures of similar path to lighting flashes. The discharging process can be carried out either through the substrate’s contact with a ground electrode or by nucleation—an instantaneous discharge cause by putting a small hole above the irradiating site, Figure 2.11.
2. LITERATURE REVIEW

Figure 2.11: 3D microvascular network creation via electrostatic discharge by: a) ground contact, b) nucleation; and c) Time-line photographs showing the process unfolding, after Huang et al. (2009).
2.5.3 Intrinsic Regenerative Materials

The constituting elements in the building block of some materials enable an intrinsic self healing function. Such material, through latent reversibility of the polymer matrix can change from monomeric state to cross-linked polymer. This congenital healing function involves formation of cross-linked polymer via thermally reversible covalent bonding and hydrogen bonding. It can also be carried out through molecular dispersion of thermoplastic additives and ionomeric coupling. The most widely accepted method of intrinsic healing is by synthesising the polymers through Diels-Alder (DA) and thermal polymeric bond reviser through retro-Diels-Alder (rDA) (Rickborn, 2004). Using additive dispersion, Zako & Takano (1999) and Hayes et al. (2007) showed self healing can be achieved using an already contained material in the matrix. Under heat application, the subordinate material particles melted, and subsequently filled internal cracks without compromising the mechanical properties of the host.

To an extent, other mechanisms such as micro-encapsulation and vascular passages of healing agent are dispensable since the migration of newly formed cross-linked polymers is heat enhanced. In addition, on board catalyst is eliminated and complex changes from accommodating the aforementioned systems to the material’s architecture along with the challenges of maintaining the mechanical integrity will become non-existent. However, the necessity for heat supply raises the question as to whether the system is autonomous in status. Conversely, prominent partisans (Chen, 2003; Chen et al., 2002) of the system advocated that the mechanism be viewed as a complete inherent package of self healing requirements. However promising the system may be, there remains a lag-time in its instantaneous response to detected damages, a conversant characteristic of other repair methods. The reviews by Wu et al. (2008) and Blaiszik et al. (2010) outlined other forms of intrinsic and other self healing methods.
2. LITERATURE REVIEW
Chapter 3

Adhesives as Repairing Agents

3.1 Introduction

Different techniques of self-healing have been discussed in Chapter 2. Regardless of the advancement in techniques, design or method deployed for any self healing application, the utmost important and essential constituting element remains the healing agent. No mechanical strength and properties recovery will be possible without a bonding mechanism that allows disjointed structural members to operate as a unique entity. The repairing agent functions like the binding matrix for FRP composite at the fabrication stage. The matrix makes singularly weak fibre to be strong as in compound fibres. Likewise, the repairing agent enables the coalescing of disintegrated members or disintegrated adjacent members with potential delamination threat to be a functional homogeneous unit and abate the possibility of drop in the operational efficiency.

There are different types of prospective adhesive for self healing application, although few researchers are concentrating on this aspect of autonomous healing yet, as the concept is still considered by many to be at the early stage. Nevertheless, it is envisaged that potential healing agent must demonstrate few important features such as; low viscosity, long shelf life, less complex polymerisation mechanism, good post-curing chemical stability, considerable toughness/strain energy - to maintain structural integrity of the host etc. Among the commercially available
3. ADHESIVES AS REPAIRING AGENTS

Structural adhesives currently in the market, only a few meet these criteria. The most likely contenders are the *moisture* activating polymerisation adhesives and *solvent evaporation* based curing methods. This chapter focuses on cyanoacrylate adhesives of moisture activating chemistry. It proceeds with a summary of adhesion theories and adhesion failure theories followed by a review of the grades of adhesives used all through this thesis. It concludes with a synopsis of other areas of the CA adhesives’ applications, especially in medical science.

3.2 Adhesion Theories

There are various stipulated theories explaining the mechanism of bond interaction between two adjacent materials, the adherends and the intermediary substance, the adhesive, Figure 3.1. It is often thought that adhesive bonding can be explained through a combination of theories. Whatever theory or combined theories governing the interlocking process of bond creation, the effectiveness of the mechanism depends on the contact surfaces of the adherends and the adhesive. Rougher surfaces are expected to produce more efficient bonding through better interlocking. This is the basis of the *mechanical theory* where the adhesive fills pores, cavities, and voids on the adherend surface. However, it has been reported that good adhesion on smooth surfaces shows bond strength development is not constrained by the surface topology of the adherends (DeMejo et al., 2000).

![Figure 3.1: Bonding Schematic](image)

Further, adhesion can be achieved by *inter-diffusion of molecules* between the adherend and the adhesive. This theory is most particularly suited to adhesion between adhesives and adherends of similar nature e.g polymer materials
3.2 Adhesion Theories

with migratable long-chain molecules which are soluble in each other. Also, the material similarity does not only influences the diffusion rate, the post-adhesion stress concentration is avoided due to the absence of any discontinuity. Similarly, physical absorption theory or the wetting theory states that the conditions for favourable joint strength is that the contact angle between the adhesive and adherends or substrates must be zero, (Ebnesajjad, 2010). In other words, every crevice must be filled for surface interfacial forces to develop between the two. In such a case, the adhesive must be of lower surface tension compare to any of the adherends (if dissimilarity exists). Otherwise, the adherend(s) surfaces can be treated to raise the critical surface energy, \( C \), and the polarity. The wetness brought by the zero angle of contact between the materials ensures that the adhesive’s molecules are in close proximity with one another and hence the Van der Waal forces between them, though small, will contribute to the strength of the joint.

Furthermore, the type of chemical bonding between the two materials also determines the mechanism for the development of the bond strength. The interaction between the liquid adhesive and the adherend depends on the prevailing molecular chemical bonding between them. The stronger the interactive forces the better the bond’s strength. Depending on the materials’ nature, the interaction level is determined by these types of bonding covalent, ionic, hydrogen bonding, and Van der Waal forces of attraction. The presence of either covalent or ion bond results into stronger adhesion compare to the latter two of which Van der Waal constitutes the weaker bond.

3.2.1 Adhesion Failure

The unified material in Figure 3.1 could experience failure along the bonded plane of interaction. This is referred to as an adhesive failure where one half of the adherend peels off with adhesive still attached to the other half, Figure 3.2a. The failure may occur within the adhesive and along its plane such that the locus of each of the failure surfaces has the imprint of the adhesive on it, Figure 3.2b. This is termed cohesive failure. If the failure is located in the adherend, such that
the adhesive molecules had forged stronger bonds with adherends’ molecules on
the surface leading to a failure within the adherend, it is called *cohesive failure in
the adherend* or *inter-adherend failure*, Figure 3.2c. These joint failure modes do
not quite explain the complex failure mechanism, which can be due to a number
of harmonising factors. Improper wetness of the surface will translate to joint
strength falling below the achievable peak strength, Figure 3.3. Likewise, differ­
ent levels of intricacies accompany the curing or polymerisation process of the
adhesives in the form of induced internal stress from expansion, contraction and
material dissimilarities. Often bonded joints fail due to unsatisfactory acclimati­sation with the operating environment.

![Image](image-url)

Figure 3.2: Failure modes of bonded adherends

![Image](image-url)

Figure 3.3: Spreading of adhesive on adherend surface
3.3 Cyanoacrylate Adhesive

Cyanoacrylate (CA), the superglue or wonder adhesive, is one of the most widely used single part, instant curing thermosetting adhesive. It is almost a “universal” glue with the capability of adhering to almost anything. This was not cherished until its rediscovery in 1958 by Harry Coover of Eastman Kodak Chemicals. Ever since, it has been found useful for domestic appliances and across many industries.

The generic polymerisation mechanism of CA adhesive requires no heating or catalyst aside the activators such as humidity (moisture presence) and hydroxide ions. When these conditions are met, sufficient bond strength is quickly developed between the adherends within few seconds of bonding. The CA grade, the bonding temperature and humidity, as well as the nature of adherend surfaces (Section 3.2), all influence the rate at which the joint strength develops. In the absence of ionic initiator, relative humidity (RH) plays a crucial role. High RH enhances faster curing but creates weaker joint. A lower RH value, leads to slow polymerisation (Petrie, 2006). However, as Figure 3.4 shows bond strength can be optimised at a RH range of 40% to 60%.

CAs are commonly associated with low viscosity making them a prospective adhesive for bonding smooth and closely spaced surfaces. It has also been found useful in clinical surgeries by surgeons and dentists. During the Vietnam War, it was used by military doctors to aid haemorrhage or blood clotting, and in bonding the skin (Murtuza et al., 2007). However, it was later stopped due to the adverse side effects on the patients before it was later rationalized through synthesizing of long alkyl chain monomers into CA compounds. The chemical structures of common CA adhesives are shown in Figure 3.5.

3.3.1 Synthesis of Cyanoacrylate Monomer

Cyanoacrylate based adhesives are formed from the homologous series of the alkyl esters of cyanoacrylic acids such that most are based on alkyl-2-cyanoacrylate,
3. ADHESIVES AS REPAIRING AGENTS

Figure 3.4: Influence of relative humidity on CA post curing joint strength, after Petrie (2006)

![Relative humidity vs Curing time graph]

Figure 3.5: Chemical structures of common cyanoacrylate adhesives

Methyl-2-cyanoacrylate

Ethyl-2-cyanoacrylate

Butyl-2-cyanoacrylate

Figure 3.5: Chemical structures of common cyanoacrylate adhesives
3.3 Cyanoacrylate Adhesive

Figure 3.5, alkyl being the homologous family. Alkyl-2-CAs are commonly produced through synthesis of an alkylcyanoacetate with formaldehyde under Knoevenagel condensation, catalysed with a weak base. The Knoevenagel reaction involves addition of hydrogen compound to carbon acid compound, usually an aldehyde or ketone to avoid unsaturated product. The resulting chemical solution is instantly polymerised to give low molecular weight of alkyl-2-CA. As shown in Figure 3.6, monomeric cyanoacrylates are further produced through thermal treatment of the resulting product of the instant polymerisation under controlled conditions (Packham, 2005).

3.3.2 Stabilization and Polymerization of Cyanoacrylate

The most common industrial CA adhesives made from different short-chain acrylate monomers are the methyl and ethyl cyanoacrylates, MCA and ECA respectively. Their use cut across many industrial applications and household products. These adhesives are produced through complex chemical reaction requiring addition of stabilizer, usually weak acidic gas like nitrogen monoxide (NO) or sulphur.
dioxide (SO₂) to keep the product in liquid form. The stabilization effect essentially prevents pre-curing action of the adhesives in their storage containers. Conversely, while weak acids aid stability, weak base, such as water or alkaline surface, initiates bond formation between the adherends and the thin layer of CA through ionic polymerisation of the monomers in the presence of hydroxide ions. The susceptibility of the adhesive to rapid polymerization is initiated by strong attraction of the $\pi$ electrons in the cyanoacrylate microstructure to the neighbouring anions and nucleophiles. Polymerization on adherends is activated by the ions present on the substrate’s surface. The nucleophile can be ions such as the hydroxyl ions in Figure 3.7. Also, the thickness of the adhesive film influences the polymerisation rate and consequently the rate of bond strength development. Example of this reaction for ethyl-2-cyanoacrylate is shown in Figure 3.7 and Figure 3.8.

**Initiation:**

\[
\text{OH}^- + \text{CH}_2=\text{C} \quad \text{CN} \quad \text{CN} \\
\text{COOC}_2\text{H}_5 \quad \text{HO}---\text{CH}_2---\text{C}^- \quad \text{COOC}_2\text{H}_5
\]

Figure 3.7: Polymerisation initiation

Ethyl-2-cyanoacrylate (ECA) and methyl-2-cyanoacrylate (MCA) exhibit notable differences in physical properties arising from their respective molecular mass. Methyl monomer CA adhesives are notably good at bonding rubber and metal while ethyl excels with plastic. Also, the monomers can be modified for specific characteristic such as low viscosity, improve thermal resistance, rubber toughened for shock and impact energy absorption etc.

The adhesives used in this research investigations are Loctite 435, and Loctite 406 (supplied by Henkel Loctite Ireland). They are both colourless liquid of 1.1
3.3 Cyanoacrylate Adhesive

Propagation - through polymer chain formation:

\[
\begin{align*}
&\text{CN} & \text{CN} \\
&\text{HO} & \text{CH}_2 & \text{C} & + & \text{CH}_2 & \text{C} & \rightarrow \\
& & \text{COOC}_2\text{H}_5 & & & \text{COOC}_2\text{H}_5 \\
&\text{CN} & \text{CN} \\
&\text{HO} & \text{CH}_2 & \text{C} & \text{CH}_2 & \text{C} & \text{COOC}_2\text{H}_5 & \text{COOC}_2\text{H}_5
\end{align*}
\]

Figure 3.8: Polymerisation of cyanoacrylate adhesives, after Petrie (2006)

Specific gravity and of low viscosity, 100-250mPa.s and 12-25mPa.s, for Loctite 435 and 406 respectively. The lower viscosity of Loctite 406 is achieved by complex monomer modifications e.g by varying the ECA proportion in each formation at the expense of reduced strength. Loctite 435 and 406 contain more than 80% and 60% of ECA respectively in their chemical formations. They both make rapid strong bonds with common substrates such as plastics and rubber, including metals for Loctite 435. These CAs are characterized by fast polymerization on adherends as shown in Figure 3.9. The CA adhesives are based on ECA chemistry which has been shown to produce stronger bonds on plastic compared to MCA (Ebnesajjad, 2010). They are also compatible with materials of thermosetting nature making CA adhesives potential healing agents in autonomous repair of microcracks in glass/carbon fibre reinforced polyester/epoxy composite structures like, WTBs, aircraft wings etc. As Figure 3.9 shows, thermosetting materials e.g epoxy, can recuperate lost strength within minutes of active repair.

However, CA adhesives are not all efficient at elevated temperature as shown in Figure 3.10. As the figures show, the optimum usable thermal condition for ef-
3. ADHESIVES AS REPAIRING AGENTS

Figure 3.9: Loctite 435: Shear strength development over curing period for different adherends at 22°C and 50% relative humidity. (Loctite, 2006)

Effective joint strength is, keeping the adherends below the flash points at 80°C - 90°C for Loctite 435, and below 80°C for Loctite 406. Further, CA adhesives with unprotected bond line can be weakened overtime and similar vulnerability is exhibited in the presence of diluted alkali solutions (Ebnesajjad, 2008; Petrie, 2006).

Cyanoacrylate Ancillary Additives

As mentioned earlier, weak acidic gases like NO and SO₂ prevent anionic prepolymerisation of the adhesive. The use of gaseous elements like these comes with the possibility of imbalanced or non-homogeneous mixture. Alternatives such as boron trifluoride is effective in this regard. Also, the addition of phenolic compound such as benzene-1,4-diol or hydroquinone, as a free radical stabilizer, improves the shelf-life and stability during transportation. The earlier version of CA adhesives with specific additives were know to be brittle showing low impact strength and peeling off tendency. The new grades of CA based on modified compatible monomers are showing improved toughness, thermal and impact resistance on susceptible substrate like plastic. The introduction of phthalic anhydride
3.3 Cyanoacrylate Adhesive

![Graphs showing shear strength over exposure time for different temperatures](image)

(a) Loctite 435

(b) Loctite 406

Figure 3.10: Shear strength of adherends subjected to different thermal conditions and tested at room temperature of 22°C. (Loctite, 2006) & (Loctite, 2012)

into CA adhesive formation has produced CAs of better resistance to heat and moisture (Petrie, 1999).

Aside the stabilizers and co-monomer additions, plasticizers are also added to address the brittle nature of the adhesive and to create flexibility along the bondline. This reduces the vulnerability to degradation (both physical and chemical). Stress develops along the bondline adjacent to both adherends due to the rapid curing nature of CA on substrates. Typical plasticizers for cyanoacrylate adhesives are presented in Table 3.1. The addition of plasticizer to CA influences the resulting bond strength. A demonstration of this effect carried out on glass substrates using dibutyl phthalate as plasticizer with n-butyl cyanoacrylate adhesive showed the bond strength decreases beyond 40% of the plasticizer concentration (McDonnell et al., 2003).

Thickeners are also added to create gel like form of CA adhesives. The increased viscosity makes such CA grades useful as structural adhesives on materials such as wood, fabrics and leather, and on materials of poor adhesion with conventional low viscosity CAs. Compounds of high molar mass like those in the second column of Table 3.1 are useful thickeners. In the case of applying CA to adherend of acidic surface, which naturally leads to retardation of the adhesive’s polymerisation, the curing process is accelerated through added accelerator such as amine,
3. ADHESIVES AS REPAIRING AGENTS

<table>
<thead>
<tr>
<th>Plasticizer</th>
<th>Thickener</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dioctyl phthalate</td>
<td>Polymethacrylates</td>
</tr>
<tr>
<td>Dioctyl adipate</td>
<td>Poly(3-hydroxybutyric acid)</td>
</tr>
<tr>
<td>Lauric acid</td>
<td>Lactic-glycolic acid copolymers</td>
</tr>
<tr>
<td>Tri(2-ethylhexyl)phosphate</td>
<td>Poly(glycolic acid)</td>
</tr>
<tr>
<td>Glyceryl tributyrurate</td>
<td>Poly(cyanoacrylate)</td>
</tr>
<tr>
<td>Diethyl sebacate</td>
<td>Poly(lactic acid)</td>
</tr>
<tr>
<td>Triethyl phosphate</td>
<td>Poly(e-caprolactone)</td>
</tr>
<tr>
<td>Dimethyl sebacate</td>
<td>Polyacrylates</td>
</tr>
<tr>
<td>Trioctyl trimellitate</td>
<td>Polyorthoesters</td>
</tr>
</tbody>
</table>

Table 3.1: Common Plasticizers & Thickeners compounds, after Leung & Clark (1996)

calixarene compounds, and silacrown compounds. This can either be achieved through adherends pre-bonding application of the accelerator to one adherend and the CA adhesive on the other, or through intrinsic application whereby the accelerator is included at the adhesive’s formulation stage. Other modifications and associated benefits have been published (Petrie, 2006).

3.3.3 Degradation of Cyanoacrylate

CA monomer at a cured or an uncured state can be broken down by pyrolysis or hydrolysis. As discussed, monomer modification through additives somewhat toughens the cyanoacrylate against thermal decomposition. However, polar adhesives such as an epoxy and cyanoacrylate can be hydrolysed via diffusion of moisture resulting to swelling and deformation with the possibility of displacing the initially rigid bond at the adhesive-adherend bond interface. The susceptibility of CA monomer to hydrolysis is attributed to the electron-withdrawing groups in the polymer. It has been reported that CA hydrolyse through an “unzipping” mechanism involving hydration of the CA polymer, an elimination of monomer unit and a reverse Knoevenagel condensation, (Cooper et al., 1989; Wade & Leonard, 1972). Aside the formation of 2-cyanoacrylic acid and formaldehydes as the by-products of the uncured and cured state respectively, unexpected
chemical properties may be displayed for the pre-cured case, while in the post-cured case, the adhesive will be blighted with weakened bond (Down & Kaminska, 2006). The reaction follows similar ester’s hydrolysis as shown in Figure 3.11.

$$\text{CH}_2=\text{C} - \text{CN} \quad + \quad \text{H}_2\text{O} \quad \iff \quad \text{CH}_2=\text{C} - \text{CN} \quad + \quad \text{ROH}$$

Figure 3.11: Possible hydrolysis of CA. Similar to hydrolysis of ester

Moisture diffuses into an adhesive joint from three fronts: through the adhesive, at the interface and through the adherend. The diffusion follows Fick’s second law as given in equation 3.1 along the x-direction (Lee, 1987).

$$\frac{\partial c}{\partial t} = D \frac{\partial^2 c}{\partial x^2} \quad (3.1)$$

where D is the diffusion constant, $c$ is water concentration, and $t$ is time. The diffusion constant would depend on the CA monomer chain, as it has been found that degradation rate is inversely proportional to the chain length. This implies that higher alkyl homologous such as butyl ester would be less prone to hydrolysis than the ethyl ester (Al-Khawam, 1983). Also, fresh CA adhesive tend to resist hydrolysis degradation compared to similar grade of older age (Down & Kaminska, 2006).

### 3.3.4 Cyanoacrylate as a Structural Repairing Adhesive

Cyanoacrylate adhesives are not known for gap filling capabilities or stability at elevated temperature above 100 degrees and hence are seldom used in the world of structural repair (Petrie, 2006). However, they have been found to possess potential as a delamination repair agent for FRP composites (Bader et al., 2000). Furthermore, in an experimental test report by Kister et al. (2007), CA showed better mechanical performance than epoxy adhesive when used on optical fibre,
under dry or wet environmental conditions. An advantage of FRP composites is the synergy of the constituting components. This subsequently depends on the proximity of the adjacent layers and fibres. Therefore, in repair of damages such as micro cracks and delamination, it is imperative to have low viscosity adhesive for maximum penetration and to retain the initial architecture of the FRP structure. This means using repairing chemicals unaffected by confined spaces which will not expand after cure. In this regard, CA adhesives can be utilised.

Several studies have reported on the use of CA for repair in an FRP structure. Dry (1996) reports CA to have efficiently repaired initiated damages in a polymeric matrix such that upon re-testing new cracks were formed. Similarly, Bleay et al. (2001) examined CA for delamination repair in FRPs. They concluded CA was unsuitable for repair due to the fact that its curing was observed to be faster than the diffusion rate out of the embedded adhesive vessel. The adhesive cures quickly due to the alkaline surface of the hollow glass fibre used. Bader et al. (2000) conducted similar experiments as reported in Chapter 5 but a durability study was omitted. They reported impressive recovered fracture toughness values for carbon fibre epoxy composites using cyanoacrylate adhesive. Among the only few durability test on FRP and CA joints, Ferreira et al. (2002) reported shear and fatigue test results for CA-FRP lap joints. Their report concluded that CA bonds degrade when immersed in water. This susceptibility to deterioration in high humidity environments is the motivation of the durability study presented in Chapter 5.

3.3.5 Other Applications of Cyanoacrylate

Higher homologous of alkyl-2-CA such as isobutyl, n-butyl-2-CA, and n-octyl-2-CA are generally considered as tissue adhesive and have been used extensively in laceration treatment. Their use holds some advantages over the traditional surface wound treatment like stitching, sutures, plastering and bandage use. The fast polymerisation rate, low viscosity means wound surfaces can be closed quickly with complete coverage thereby diminishing the chances of infection and secondary haemorrhage, which often results from stitching. Also, hypertrophy and
3.3 Cyanoacrylate Adhesive

scar formed from plastic surgeries can be curtailed using tissue adhesive and the 
application is also time efficient. Although, the associated heat of polymerisa-
tion remains a concern to the functioning of circumscribed tissue. Butyl ester 
cyanoacrylate is commonly used in the treatment of small wounds, bone fracture 
and vascular surgery or tissue closure. On applying it to modelled skin wound, it 
was found to cause less tissue irritation through less inflammation (Ebnesajjad, 
2010). This adhesive is preferable to shorter polymer-chains ones (methylys and 
ethyls) because of its slower and mild exothermic curing process. It degrades at 
lower pace compared to either MCA or EGA, such that the body is allowed an 
ample time to neutralise the toxic by-products of the chemical breakdown.

On the other hand, octyl-2-CA is one of the best cyanoacrylate brand of medical 
adhesives used in closure of surgical incisions and serious laceration (Handschel 
et al., 2006; Silvestri et al., 2006). Its higher homologous position creates longer 
chain, which enables stronger bond between its adherends. It is also more flexible 
and forms better bond than butyl-2-CA. In the 1970s, long chain cyanoacrylate 
adhesives were used in oral surgery particularly in dentistry, for post-extraction 
dressing and as blood clotting agent for severe bleeding. However, the associated 
failure of the CA adhesive at cement-dentin interface and development of better 
alternatives has led to their seldom usage over the past decade (Harod, 1990).

Cyanoacrylate compositions are also employed in cosmetics industry for mani-
cure. Nails can be damaged during manicure as there may exist imbalanced 
moisture content in the treated nails. This may lead to nail chapping underneath 
the manicure addressing. Addition of cyanoacrylate to the manicure composition 
or formulation ensures healthy nail growth as chapping is prevented due to quick 
curing and repair is almost instantaneous (Fink, 2005).
3. ADHESIVES AS REPAIRING AGENTS
Chapter 4

Preliminary Experimental Investigation of Composite Structures

4.1 Introduction

In Chapter 2, a review of laminate theories and self repair techniques were presented. Thereafter, the use of repairing agents were discussed in Chapter 3 for healing damaged structures. For developments on self healing system, it is thought to be advantageous to locate the most vulnerable areas within a composite structure. This chapter explores how and why composite structures, including wind turbine blades (WTB) fail through some preliminary experimental tests. The chapter begins with failure mechanisms of thin-walled structures. It further reports a diagnostic test on a 6 kW WTB, under a flap-wise motion for strain mapping. The mapping reveals the strain magnitudes at certain intervals across the length of the blade. It ends with a finite element analysis of the test.
4. PRELIMINARY EXPERIMENTAL INVESTIGATION OF COMPOSITE STRUCTURES

4.2 Wind Turbine Blade Failures

4.2.1 Local Stress Concentration

WTBs are complex and meticulously crafted structures birth out of engineering compromise to create stronger and lighter blades and can therefore be highly susceptible to local stress concentration. To determine the extensive influence of this phenomenon, full scale destructive tests are usually carried out. However, such comprehensive tests are often privately conducted, regarded as developmental research on wind energy and as a result, few studies have been published on the testing of WTBs to failure. Marn et al. (2009) examined a damaged blade of a 300 kW wind turbine. It was found that deep seated flaws and damages like inter-laminar separations - delamination, cracks and defects from the fabrication process played a prominent role in the damage event. The problems arising from inconsistencies in the manufacturing process often include resin deficiency areas, abrupt change in laminate thickness, misalignment and fibre disorientations. WTBs are structurally analysed as a cantilever beam, subjected to a varying load (wind), reversal and non-uniform UDL loading (self weight). Due to these uncertainties and the complex loading conditions, WTBs have high tendency of developing stress concentration at the root section, where the bending moment is maximum. At the start of this section, is the maximum chord length, the blade’s connection to the steel hub and the smallest angle on the blade profile. A design flaw in this region creates a local stress concentration that grows over time while fatigue sets in. A sharp change of angle around the maximum chord length causes re-entrant corner effects and gives rise to irregularities in “stress distribution” Timoshenko (1941). Also, change in laminate thickness causes load transmission eccentricity. This induces bending moments thereby adversely affecting the effectiveness and stability of the laminate operations Marn et al. (2009). Misalignment of laminae is equally undesirable and produces a detrimental effect particularly for the spar’s compression flange.

Research carried out on blades (Overgaard et al., 2010; Shokrieh & Rafiee, 2006; Sundaresan et al., 1999) has shown that a critical zone exists on WTBs often
4.2 Wind Turbine Blade Failures

accompanied with high probability of failure. As a result, a WTB is more likely to suffer damage in between the root section, and the quarter to third length of the blade's span, Figure 4.1. Around this region WTBs undergo a rapid change in their cross-sectional area. This section contains a huge portion of the spar, which controls the structure's ultimate strength. Sundaresan et al. (1999) recorded buckling failure of the spar web preceded by delamination at a location 37.5% along the blade from the root. Shokrieh & Rafiee (2006) also found that a critical zone lies in compression flange on the spar. The presence of cracks and delamination irrespective of their size or type in this region makes a vulnerable structure, highly prone to sudden failure by rapid disintegration while it is under operation.

Figure 4.1: Blade failure closer to the root. Failure raises safety issues.

4.2.2 Brazier Effect

Damages often result from the failure of the load carrying structure within the blade, i.e. the spar - a structure that accounts for 80% of the load while the aerodynamic aerofoil shells accommodate the remaining 20%. In recent times, various workers have attempted to explain the principle(s) governing local stress concentration development. Jensen et al. (2006) reported that the Brazier effect and Ovalisation governs blade failures. Using a static test procedure, they found

(a) (www.betterplan.squarespace.com) (b) (Courtesy of Raoul Dixon/North News & Pictures)
that the top compression flange of a WTB’s spar had ovulated leading to the separation of the aerodynamic suction shell from the flange. This also led to delamination initiation followed by buckling.

While Overgaard et al. (2010) agreed to the presence of the Brazier effect but downgraded the effect as the major cause of failure in a similar flapwise moment static test. They found that delamination and buckling played a more prominent role. They explained that the ultimate strength of the flange fibres was exceeded by localised non-linear bending strains resulting in interlaminar fractures and buckling, preceding a fatigue assisted failure.

Brazier effects (Brazier, 1927) account for the non-linear variation in strains of a material which are not considered in a simple beam theory (SBM). In SBM, a linear relationship is assumed between the bending moment $M$, and the curvature $k$ while other parameters, like the elastic modulus, $E$ and second moment of area, $I$, are kept constant.

$$M = EIk$$

Figure 4.2: The Brazier effect

Under the influence of a bending moment, a thin-walled structure of hollow circular or rectangular cross-section will flattens along the originally curved path. The deformed structure takes an oval shape profile for the circular section while the
4.2 Wind Turbine Blade Failures

square shaped object is sucked-in, Figure 4.2. These types of deformations induce strains because of the changes to the cross-sectional areas and consequently compromising the stiffness governed by $I$. As the curvature $k$ increases, $I$ experiences huge reductions, (Knaster et al., 2001). Coupled with fatigue loading, the object progressively fails under crushing pressure from the Brazier effect as the strain limit is gradually exceeded. Non-linear strain variation occurs due to the blade stiffness or resistant to increasing curvature from bending.

On a profound level, deformation in the cross-section of a WTB can be generated by internal forces under normal operation. This creates an insidious effect particularly on the aerodynamic shell and the spar due to their casting shape. The blade experiences this effect during normal operation due to its self weight and under flapwise loading at various frequencies. Internal forces in transverse and longitudinal directions initiate the cross-sectional flattening when deformation occurs from bending. Although, the transverse component of the applied forces has a more damaging effect on the spar box (Jensen et al., 2012). The moment capacity of WTBs are tested in flapwise and edgewise directions as part of the certification procedure. The flapwise direction is of particular importance; from aerodynamic lift loading, the internal forces are initiated thereby generating a flapwise moment and shear force action. Series of these actions cause web buckling, ovulation, delamination and buckling in the compression flange.

4.2.3 Delamination and Buckling

There are also other mechanisms which play more prominent roles than the Brazier effect as shown by Overgaard et al. (2010). Delamination at the critical zones and buckling of the spar's web play important roles in a blade damaging event. For a laminated FRP structure, out-of-plane stresses either developed through the normal operation (structure's functionality) or through flaw stimulated damages causing the most critical layer to fail and/or delaminate. Interlaminar failure arises due to out-of-plane stresses, $\sigma_z$, $\tau_{xx}$, and $\tau_{zy}$ as discussed in Chapter 2. The magnitudes of these stresses are considerably higher at the surfaces of adjacent layers compare to their associated values within the layer plane. Cracks develop
as a result on WTBs and quickly propagate under varying degrees of dynamic loading. Delamination then occurs at the crack front between adjacent layers irrespective of the degree of their damage state or condition. Delamination has been noted by many studies to be a chronic effect of WTBs especially on the high pressure side, (Jensen et al., 2012; Mandell et al., 2003). The order of failure is not precisely clear at present. Some evidence suggests that delamination in the compression flange near the root section causes sharp increase in load that exceed the spar’s web buckling capacity.

Delamination and buckling often occur harmoniously. Often the former is preceded by the latter. FRP laminate under critical compressive loading above its ultimate strength will be deflected out of plane resulting in failure when the deflection is unsustainable. It is quite understandable how this can cause rapid progressive failure. Delaminated laminate loses its unique homogeneous quality and strength, adversely reducing the capability of restraining or moderating the out of plane deflection.

4.3 Static and Dynamic Test on an FRP WTB

Based on the knowledge provided in the overview of possible failure mechanisms of thinned walled structures, it becomes structurally essential to validate this knowledge on an actual WTB. As described in the preceding chapter and with the accompanying illustrated images in Figure 4.1, WTBs tend to structurally fail at critical sections of least resistance against internally induced stresses irrespective of their mechanical performance certification. The objective of the test outlined in this section is to construct a stress profile of a typical medium sized 6 kW WTB and to compare and validate the resulting plot with a finite element model of the structure.

A scaled down WTB certification tests comprising of static and flapwise (dynamic) loading are used in capturing the stress profile on the blade. A full scale certification examination approves WTBs to be of required standard in strength,
rigidity and reliability all through the service life provided for in the design. Usually a full certification test that complies with international standards, such as the International Electrotechnical Commission IEC61400-23, anchors their accreditation of a blade on its performance in static and fatigue tests. These are the prominent test specifications. Generally, static tests are designed to ascertain that a blade possesses the structural necessities for normal operations and resistant against extreme load cases. For example, upscale mega watts turbines (2-5 MW, and higher) are designed with blades to withstand 50 year gust wind. In a typical static test, the blade is fixed to a makeshift hub and load is statically applied in one direction. The load can either be vertical or horizontal about the blade’s axis, in flapwise and edgewise directions to record the strain and deflection responses to the applied load. The load can be distributed across different sections of the blade using a whiffle tree/saddle method, Figure 4.3, or the load can be independently applied at the certain sections using multiple actuators as shown in Figure 4.4. Also, progressive loading can be applied as shown in Figure 4.5, which allows multiple or single test cases to be executed at single station, as well as obtaining higher shear loads.

As WTB fatigue test is not considered in this thesis, a short account of the test is provided. Fatigue test on WTB is an accelerated simulation of its normal operation through the service life. Sinusoidal loads of similar normal operational amplitude are usually applied. The load spectrum may contain 1 to 10 million cycles. The dynamic test described in this section is an extended static flapwise motion of the blade. In field, the flapwise movement of blades can be considered as a dynamic reaction to wind loading, especially for stationary blades. The influence of this motion on WTB damages was highlighted in a study by Ronold & Larsen (2000). They conducted a reliability study of blades failure due to flapwise bending under normal operating conditions. The dynamic test in this section reports on similar effects of the flapwise bending of a WTB. It examines the stress and strain development, particularly around the root section, when the blade is subjected to sinusoidal and simulated wind load excitations.
4. PRELIMINARY EXPERIMENTAL INVESTIGATION OF COMPOSITE STRUCTURES

Figure 4.3: Static test: Three-point whiffle-tree, after (Choi et al., 2012)

Figure 4.4: Static test: Use of multiple hydraulic actuators – NREL
4.3 Static and Dynamic Test on an FRP WTB

4.3.1 Properties of the 6 kW blade

The Twintex thermoplastic blade measures 2.5 m in length and has the composition of glass fibre reinforcements in a polypropylene matrix. Generally, technical data on WTBs are considered sensitive and confidential. There was no exception for this 6 kW WTB. This means, information on both relevant materials and mechanical properties of the blade was unavailable at the time this test was conducted. However, wind turbines operating with this type of blade are capable of generating around 8000 kWh annually, at 5 m/s wind speed with cut-in speed of 3.5 m/s, and can be operational at class two wind speed of 59.5 m/s.

4.3.2 Test procedure

The 6 kW WTB (EireComposite, Ireland), supported at the root section by a specially fabricated steel support base, was mounted horizontally on a test rig as shown in Figure 4.6. The normal blade-root section’s connection to the hub on the nacelle was replicated by the fabricated steel casing. The blade’s end was
fixed to the makeshift-hub support at 45 degrees, bolted at the top and bottom against a timber cushion material, which prevents the bolts from damaging the blade. The loading head of the structural testing system (STS) actuator was placed at 55% of the blade span. This in effect, allows a sizeable differentiation of the strain magnitude for the critical sections. The testing facility, STS, provided the opportunity of applying a simulated wind load for a bandwidth with a frequency range. Also, an effective management of the operation is provided in the relaying back of the command signals. This allows a relative comparison between the input command (force/displacement) and the feedback information - the real force and displacement magnitude applied to the blade. For better precision and reduced ambiguity, the test was conducted using the displacement command due to the staggering size of the 160 kN load cell actuator to the 6 kW WTB. In order to ensure an even load distribution across the width of the blade, at the point of contact with the STS actuator, the circumferential profile at 55% of the blade span was mapped and sculptured unto a timber plank. This allowed the actuator head to be aligned horizontally and parallel to the blade.

Figure 4.6: 6 kW WTB static test set-up

The blade was instrumented with twelve 120 Ω, 1500 mV (capability) aluminium foil strain gauges (Radionics, RS 632-168) along the centreline of the blade's length. Before strain gauges were attached their situated positions were slightly
scored with sandpaper and cleaned with acetone. The strain gauges were held in position using Loctite 454 super glue. The strain gauge placements were divided into two sections, one section on either side of the actuator. This was done for both the compression-suction side (top) and the high pressure (bottom) side. The first section consisted of four evenly spaced strain gauges at 160 mm apart from the hub connection to the actuator. In the second section, two more strain sensors were placed at same the spacing after the actuator. A clearance of 160 mm was allowed for the nearest gauges to both the steel hub and the actuator.

The gauges were labelled G1-G6 for the suction side and G7-G12 for the bottom side, Figure 4.7. Sensors G1 & G7 are the closest strain gauges to the hub, and G6 & G12 are the furthest. The maximum deflection of the blade was measured at the tip using a mounted tipping/dropping head of a linear variable displacement transducer (LVDT). All data from the twelve gauges and the LVDT were collected via a computer’s automated data acquiring system - Strainsmart. The blade was tested dynamically, in the flap wise direction for two different amplitude of 4 mm and 8 mm for 0.5 Hz sinusoidal wave. The second dynamic test uses white noise, a representation of wind loading, using a bandwidth frequency of 0.5 Hz to 3.5 Hz and a root mean square (RMS) value of 8 mm.

Prior to the commencement of the dynamic test, the blade was statically loaded like a cantilever beam. The load was applied (in increments of 10 N up to 190 N) at the actuator’s position by virtue of a load hanger. Readings were taken for every load increment.

4.3.3 Results and Discussion

Static test

The results in Figure 4.8 and Figure 4.9 show the peak strain to be emanating from the second strain sensor from the root on both sides of the blade. Figure 4.8 compares the strain magnitude from each sensor from the tension side as the
Figure 4.7: Schematic layout of the blade showing the strain sensors' positions
static load increases. The strains from the compressive side of the blade (bottom sensors) show similar profiles to those at the top, as Figure 4.9 shows. In Figure 4.8 and Figure 4.9, the peak strains are stationed around strain sensors G2 and G8. This directly translates to the highest stress areas according to the stress and strain relation. This shows that the blade exhibits permanent feature of vulnerable area prone to high stress than any other part on the blade. The blade's responses shown in the figures will be replicated in an event of birds striking, falling hail-stones, and snow build-up on the blade when it is non-operational. Consequently, the outcomes such as those shown in Figure 4.1 will be inevitable.

![Figure 4.8: Relation between the strain and the applied load for the blade's top side](image)

Furthermore, in Figure 4.8 strain gauge, G6, appears to be isolated, showing negative strains as the load increases. Figure 4.9 also illustrates that strain gauge G12 replicated similar response, simultaneously as the load increases. This is an
4. PRELIMINARY EXPERIMENTAL INVESTIGATION OF COMPOSITE STRUCTURES

Figure 4.9: Static test: Normalised strain profile along the 6 kW WTB

unexpected occurrence for a cantilever structure. The bending moment beyond the actuator should read zero Nm, like the strains. This exception can be attributed to the possibility of surface stresses developed around the gauges due to relative proximity to the actuator.

Dynamic Test

Prior to the dynamic test, the blade was transiently loaded to determine the non-destructive dynamical loading window of frequencies. It is imperative to avoid damaging the blade through resonance at its natural frequencies during the proposed test. Figure 4.10 shows the strain reading of the transient load around the root section, and a subsequent plot in the frequency domain. The latter plot of Figure 4.10 shows a peak amplitude at approximately 5.85 Hz. This analysis was performed as it is necessary for the blade to stay clear of its first flapwise natural
frequency and operate within a less damaging frequency range for the dynamic test.

![Figure 4.10: Free vibration of the WTB: a) Associated strain reading. b) Frequency response](image)

The plots of the peak strain at each gauge location along the blade are shown in Figure 4.11 and Figure 4.12. Neighbouring legends do not necessarily attain their respective maximum strains simultaneously. As the actuator completes a sine wave period, the blade experiences alternating tensile and compressive stresses, which are instantly picked up as strains in the outer surface by the strain sensors. Due to the elasticity relation, the level of strain directly corresponds to the stress magnitude induced in the blade. It can be seen from the two plots that the top half of the blade has more stress attributed to it when compared with the bottom side. Taking Figure 4.12 as a reference, both the tension and compression profiles appear like an inverted mirror reflection of each other. It can be observed from the Figure 4.11 and Figure 4.12 that as the amplitude increases so does the strain
magnitude at each sensor location along the blade. The upper half of the blade was subjected to 45% stress-strain tension and 19% in compression more than the bottom half. This can be attributed to the presence of more curvature on the upper surface than the lower surface according to the blade’s cross-sectional aerofoil profile. The latter numerical figure does not necessarily account for sufficient stress accumulation for WTB damages. However, the presence of such minuscule stress could catalyse other destructive factors, such crack propagation, fibre-matrix debonding, delamination, buckling etc.

Figure 4.11: Peak strain values along the blade for 0.5 Hz sine wave with 4 mm amplitude

The maximum strain was recorded around 20% of the blade’s length. This location can be associated with the local stress region. Figure 4.13 shows the strain plot from the white noise excitation for two segments at 0.2 R and 0.5 R. The peak strain plot at each gauge location is analogous to Figure 4.11 and Figure 4.12. The white noise excitation shows the magnitude of the strains at a sensor
location closer to the loading point and at a location considered as a structural hotspot. The magnitude of the strain at 0.5 R can be observed from the plot to be approximately 12.5% of that at 0.2 R. Although it can be argued that since it is a cantilever structure, the influence and magnitude of the reaction forces at the support will dwindle down the structure’s length, hence higher deflection at such a point. However, this theoretical assumption leaves a sceptical view in that the highly stressed portions should be in proportion to the bending moment, which should be at the base, or the support, or closer to strain gauge G1. This was not the case.

4.3.4 Finite Element Analysis

A finite element model analysis of the blade was carried out using Ansys Mechanical 11.0. The blade’s dimension details were measured for the computer model construction. The model was statically analysed. Keypoints at definitive curva-
4. PRELIMINARY EXPERIMENTAL INVESTIGATION OF COMPOSITE STRUCTURES

![Strains at two different sections of the blade in response to white noise excitation](image)

Figure 4.13: Strains at two different sections of the blade in response to white noise excitation

Ture points along the blade were joined together forming lines using the spline command and followed by area generation. The model was 2100 mm long and 500 mm wide at the root. The thickness of the hollow rectangular root was 5 mm.

Shell element elastic 281, an 8 node-element with 6 degrees of freedom (UX, UY, UZ, ROTX, ROTY, and ROTZ) at each node, was chosen for this analysis. The element operates on first-order shear deformation theory (FSDT). The choice was informed based on the element’s range of capabilities such as laminated composite shell analysis and compatibility with non-linearity. The exact fibre layering configurations or angles along with vital mechanical properties of the actual blade were unknown for this modelling. However, previous literature (Corbyn & Little, 2008) had shown that medium size urban FRP blades similar to the tested blade usually have cross-ply clothing fibres of alternating $[0°/90°]_n$ which are then ori-
4.3 Static and Dynamic Test on an FRP WTB

Oriented in $[45^\circ]_n$. This was inherently employed as the layer or ply stacking order. Using shell section input, $[\pm45/0/45/0/\pm45]$, 7 layers of 0.71 mm thickness per layer was used. Two layers of $[0^\circ]$, oriented parallel to the principle axis, were added to further stiffen the model. The orthotropic elastic properties used in the model are listed in Table 4.1.

<table>
<thead>
<tr>
<th>$E_x, E_y = E_z$</th>
<th>100, 6.88 GPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>$PR_{xy}, PR_{yz}, PR_{xz}$</td>
<td>0.201, 0.296, 0.201</td>
</tr>
<tr>
<td>$G_{xy}, G_{yz}, G_{xz}$</td>
<td>3.605, 3.03, 3.605 GPa</td>
</tr>
</tbody>
</table>

Table 4.1: Laminate properties

The blade model was map-meshed into equal quadrants of 50 mm in size. A zero displacement constraint was placed along the root line section to act as a support for the cantilevered blade. Similarly, a downward pressure, equal in magnitude to the maximum load from the STS hybrid actuator was exerted on the model at 55% of the blade span.

4.3.5 FEA Result and Discussion

From Figure 4.14a, up to a quarter length of the blade root is depicted to be of high strain region, hence high stress. This is in agreement with the region of high strains in the actual experiment. However, some points in the red shaded region are of different level of significance to one another in relation to their stress intensities. The principle used to categorise the most vulnerable section is the chosen failure criteria option, which is based on the material strength. There are few common principles: Maximum Stress, Maximum Strain, Tsai-Wu, and Tsai-Hill—the Maximum Work theory criteria.

The first two criteria operate on equality principle by comparing the given ultimate material strength against the induced stress or strain at each node or for
4. PRELIMINARY EXPERIMENTAL INVESTIGATION OF COMPOSITE STRUCTURES

Figure 4.14: Stress & Strain Plots from FEA

(a) High Strain Locations  (b) Tsai-Wu Stress Plot

each elemental unit. From a theoretical perspective, the latter two of the criteria have the head-on advantage in that they provide the prospect of obtaining a material strength, using the maximum work theory in particular, and the output results are influenced by strength interactions, a feature that is non-existent in either the Maximum Stress or Maximum Strain theory. The Tsai-Wu criterion defined in the Ansys software package was used in the analysis by inputting the critical strength parameters for the blade model. The model allows the solver to grade the severity of each element or at every node. The value of the strength parameter for which failure is not expected is greatly increased. In that case, the resulting stress, or strain, or shear strain, will be below its corresponding critical value.

Figure 4.14b, shows the output stresses. The severity level of similar stress regions are marked in colours, 'red' denotes failure hotspots and the 'blues' are serenity zones. The Ansys provided Tsai-Wu criterion was used for the blade with the following values:

<table>
<thead>
<tr>
<th>Stress Type</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stress in tension (MPa)</td>
<td>1830(x), 57(y), 105(z)</td>
</tr>
<tr>
<td>Stress in compression (MPa)</td>
<td>-1096(x), -228(y), 105(z)</td>
</tr>
<tr>
<td>Shear stress (MPa)</td>
<td>71(xy), 1000(yz), 1000(xz)</td>
</tr>
</tbody>
</table>
The normalised strain plot of both the experimental and the finite element analysis is shown in Figure 4.15. The experimental plot in Figure 4.15 comes from the tension side plot in Figure 4.9. A normalised plot was chosen to enable proper comparison between the two set of results. The strains from the FEA show similar high strained areas with similar variation along the blade’s length compared to the experimental result, though there are some differences in magnitude. The differences can be attributed to the orthotropic elastic properties used. Therefore the comparison plot in Figure 4.15 is based on the normalised strain from the experiment. As both plots show, they seem to have the peak or highest strain value around the 20% of the blade’s length from the root and a similar slope descending from the peak value.

![Normalised Strain](image)

Figure 4.15: Normalised strain plots for the experimental (Figure 4.9) and FEA
4. PRELIMINARY EXPERIMENTAL INVESTIGATION OF COMPOSITE STRUCTURES
Chapter 5

Experimental Investigation on Delamination-Interlaminar Fracture

5.1 Introduction

In Chapter 3 the use of repairing agent with a particular focus on cyanoacrylate for adhesion and bonding was discussed. Chapter 4 presented some preliminary investigations on the development of stresses and failure mechanisms in FRP composite materials. Generally, FRPs are considered inert, hence their wide usage, even in marine and saline environments. The same cannot be said of FRPs with cyanoacrylate adhesive joints. There are few published literature in this regard. This chapter experimentally investigates the potentials of a cyanoacrylate adhesive when used as a repairing agent in an FRP self healing system, especially for an off-shore structure. The prospect of the adhesive is investigated through the determination of the fracture toughness, $G_{IC}$, of delaminated glass fibre polyester composites bonded with cyanoacrylate adhesive under ambient conditions. It also studies the influence of environmental exposure of GFRP coupons bonded with the adhesive on their resistance to delamination.
5. EXPERIMENTAL INVESTIGATION ON DELAMINATION-INTERLAMINAR FRACTURE

5.2 Fracture in Composite

The destructive prowess of delamination in FRP composite structures has been explicitly established in many studies as discussed in Section 2.4. It has equally been shown that further enrichment of crack propagation to delamination can be aborted through self healing application. It is not adequate to activate the self healing system without assurance of multifaceted restoration of the mechanical properties, in terms of the recovered bending stiffness and the intermediary toughness between the bonded adherends or substrates. The repairing chemical should be an effective surrogate to compliment the ineffectual matrix. Structures including wind turbines are increasingly sited at locations renowned for their harsh climate and any repair agent must be durable under such conditions. One of the advantages of FRP composites is their environmental durability and chemical inertness. However, the reinforcing fibres, similar to reinforcing steels in concrete, are vulnerable to chemical attack, although the surrounding polymer matrix offers some protection. Some WTBs have been reported to have failed due to lightning strikes. A primary factor leading to this failure is water penetration through an open crack on affected blades thus creating a conductive path channelling the lightning into the blade (Caithness, 2011; Kithil, 2008). Therefore, in the event of carrying out a repair work, it is essential to ensure that structures exhibit resistance to highly probable moisture ingress.

This chapter demonstrates how effective a cyanoacrylate (CA) adhesive can be in repairing and delaying delamination when it is employed as a repair agent in a glass/polyester composite. A study conducted prior to this experiment showed the vulnerability of CA adhesives to certain degradation elements as outlined in Section 3.3.3. The test outlined in this chapter examines the environmental integrity of a CA bond following chemical, and temperature exposure. Composite samples were prepared in-house and subjected to a Mode I (opening) double cantilever beam (DCB) test. The aim of the experiment was to determine the fracture toughness of the repaired composite, which contains a CA layer, with respect to the initial interfacial toughness of the glass/polyester composite.
5.3 Experimental Procedure

5.3.1 Preparation of Samples

Two sets of specimens were fabricated. The first set, made using 200 g/m$^2$ woven E-glass, was designated for DCB testing under ambient conditions. The second sets were fabricated with 690 g/m$^2$ unidirectional E-glass fibres. These were specialised for an environmental impact assessment of FRP composites with respect to influence of CA adhesive on their mid-plane. Table 5.1 illustrates the characteristics of each set and the shared method of specimen preparation is outlined in subsequent paragraphs.

<table>
<thead>
<tr>
<th>Set</th>
<th>Ambient</th>
<th>Environmental</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mould size (mm$^2$)</td>
<td>288 x 210</td>
<td>300 x 300</td>
</tr>
<tr>
<td>E-Glass fibre mass (g/m$^2$)</td>
<td>200</td>
<td>690</td>
</tr>
<tr>
<td>Number of Layer</td>
<td>10</td>
<td>6</td>
</tr>
<tr>
<td>Mass of glass fibre (g)</td>
<td>86.60</td>
<td>122</td>
</tr>
<tr>
<td>Polyester resin (g)</td>
<td>216.5</td>
<td>305</td>
</tr>
</tbody>
</table>

Table 5.1: Two sets of fabricated specimens and their constituting properties

A polyester/glass fibre composite was chosen for this research due to the ease of supply, the relatively low cost, and also it is the most widely used material by the WTB manufacturing industry. In 2001, an estimated 50 million kilograms of glass fibre laminates were used in manufacturing WTBs (Griffin & Ashwill, 2003). For this investigation, a polyester matrix was produced using a mixture of Norsodyne H 13212 TAE orthophthalic polyester resin (Glass fibre and Resin Supplies Ltd, GRS) with Metox-50 - methyl ethyl ketone peroxide (MEKP) as the curing agent (Oxytop Ltd). The matrix formulation was based on the manufacturer’s recommendation of 2:100 weight ratio and the addition of MEKP at 2% of the resin’s
weight. E-glass fibres (GRS) were used as the reinforcements. The matrix was quantified on supplier's instruction at a ratio of 2.5:1 by weight of polyester resin and the glass fibre respectively. A specially fabricated aluminium mould (Table 5.1) of 10 mm depth was used to cast the laminated composite. Appropriate layers of glass fibre mattings were cut to fit their respective mould dimensions as in Table 5.1.

The mould was carefully coated with honey wax (Speciality Products), a de-moulding agent to facilitate post-curing removal of the laminate. This was followed by spreading a thin layer of the freshly mixed matrix on the mould using a 25 mm paint brush. One fibre mat was then carefully placed on the sticky matrix. This sequential process of spreading the matrix followed by placement of a fibre mat was repeated up to the mid-layer (half of the total number of layers) of the glass fibre mat. At this point, 15-µm of aluminium thin foil (Raytex brand), previously waxed with the de-moulding agent was placed along the length of the laminate at the edge of the mould. The foil extended 50 mm towards the centre of the laminate at the mid-plane creating an unbonded section, which acts as a crack initiation point for delamination propagation. The hand layup fabrication process was resumed and continued up to the last fabric layer.

The laminated panels were left in the mould to cure at room temperature for 24 hours after which they were removed. They were cured for a further 24 hours under the same conditions. Using a 5 mm mitre saw, the 'Ambient' set panel was cut into six small coupons with dimensions of 140 mm long x 25 mm wide for delamination testing. Similarly, the 'Environmental' designated panel was divided into sixteen smaller coupons of the same dimensions. Cyanoacrylate adhesive, Loctite 435 (Henkel), was used to bond two piano hinges to each coupon. The piano hinges were placed at the end of each coupon above the aluminium foil (Kessler & White, 2001). The hinges were machined to the width of the specimens to facilitate load transfer during the DCB test.
5.3.2 Application of re-bonding agent

After the first DCB test had been performed for each coupon the delaminated halves, henceforth termed adherends, were bonded together using Loctite 435, thus repairing the coupon by replacing the initial polyester bond with cyanoacrylate. On one half, 0.33 ml of Loctite 435 was evenly spread using an applicator to form a thin layer. As discussed in Chapter 3, CA polymerisation process can be achieved by atmospheric moisture or hydroxide ions contact with the adhesive, which made it suitable for this experiment (Loctite, 2006). The adhesive was applied 5 mm beyond the aluminium foil. This ensured that the bonding agent does not ingress into the initial pre-delaminated length when the adherends are bonded together. The 5 mm gap was further protected using a 16 μm wax paper. The re-bonded specimens were then held together firmly with a clamp to aid proper adhesion. The bond was allowed to cure for twenty-four hours after which the specimens designated for environmental simulated conditions were divided into four groups of four. Three of the groups were assigned to different specific impact conditions outlined in Table 5.2, kept in the conditioning chambers at the Henkel Ireland R&D facility, while the fourth group was kept at ambient temperature and pressure as control samples. The specimens' labels were inscribed on both sides of each specimen for proper identification during testing. Table 5.2, shows the environmental conditions to which each of the groups were exposed and the nomenclature used hereafter. The numeric subscript identifies individual specimen within a group.

5.3.3 Test Setup with Specimens

In this mechanical test, the BS ISO (15024:2001) (BSI, 2001) was used for guidelines as it describes the Mode I DCB test to determine the interlaminar fracture toughness. The specimens were mounted on a Zwick Roell 1474 mechanical testing machine with a 10 kN load cell. The test was conducted in a displacement control mode at a rate of 5 mm/min. Prior to the testing of the repaired specimens, their edges were cleaned to prevent cured CA adhesive bridging across the
edges which would create additional stiffness thereby complicating the analysis of the CA/polyester bond fracture toughness. Figure 5.1 shows a specimen under test, illustrating the test set-up of the schematic shown in Figure 2.3. The edges of the specimens beyond the crack initiation area were marked with a felt tip pen at 5 mm intervals over a 60 mm length. These vertically oriented lines provided a means to correlate the load with crack propagation distance. The corresponding load, \( P \), and cross-head deflection, \( \delta \), (distance between the two vertical loading pins) were recorded by the Zwick Roell’s TestExpert software and correlated with crack propagation distance which was recorded by a Panasonic HDC-TM80EB video camera with 30 frames per seconds capability.

The data from both acquisition sources were synchronised by starting the test on the Zwick and recording the test time clock. This was achieved by pointing the video camera at the PC monitor before focussing it on the specimen under test. This ensures more accuracy in correlating the measured values for the pair of transverse load and displacement with the corresponding longitudinal delamination propagation on the test specimens. This technique was used in tracking the crack length, \( a \), as it grows. Manual methods of recording data have been used by other researchers and are reported elsewhere along with photographic analysis of stick-slip delamination behaviour (Bader et al., 2000). Inspired by

<table>
<thead>
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<th>Group</th>
<th>Description</th>
<th>Condition</th>
<th>Duration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ctrl(_1)-(_4)</td>
<td>Control specimens</td>
<td>Room temperature</td>
<td></td>
</tr>
<tr>
<td>Cold(_1)-(_4)</td>
<td>Cold Test</td>
<td>-20 degrees</td>
<td>1000 hours</td>
</tr>
<tr>
<td>NS(_1)-(_4)</td>
<td>Cyclic spraying without salt</td>
<td>40 degrees, 98% relative humidity (RH)</td>
<td></td>
</tr>
<tr>
<td>S(_1)-(_4)</td>
<td>Cyclic salt spraying</td>
<td>35 degrees, 100% RH, 5% salt</td>
<td></td>
</tr>
</tbody>
</table>

Table 5.2: Specimen groups
these techniques, a similar technique using an optical scanner was used for this study to analyse surfaces of the delaminated halves of the specimens. After total delamination had occurred, an optical scanner was used to capture the images of the adherend surfaces. The images were analysed and were used to highlight the areas of exposed fibres providing a means to assess the CA coverage and its effectiveness in bonding the adherends.

Figure 5.1: Actual test setup

5.3.4 Analysis of Mode I DCB test

The analytical approach applied to the splitting mode of delamination is derived from linear elastic fracture mechanics. This implies experimental data requires a correction factor due to possible rotation as the loading point is not a built-in support (BSI, 2001). The resistive force or bond between the adjacent plies in the mid-plane determines the strain energy released as delamination propagates. This is equivalent to the fracture toughness, $G_{fc}$, of the respective specimen based on corrected beam theory (CBT) given by slight modification to equation A.8 in
5. EXPERIMENTAL INVESTIGATION ON DELAMINATION-INTERLAMINAR FRACTURE

Appendix A:

\[ G_{ic} = \frac{3P\delta}{2B(a + \Delta)} + F \]  \hspace{1cm} (5.1)

with

\[ F = 1 - \frac{3}{10} \left( \frac{\delta}{a} \right)^2 - \frac{2 \delta l_1}{3 a^2} \]  \hspace{1cm} (5.2)

where \( P \) (N) is the applied load; \( \delta \) (mm) is the corresponding load line displacement; \( F \) is the large displacement correction factor; \( b \) (mm), is the specimen width; \( h \) (mm) is the thickness of one half of the substrate; \( a \) (mm) represents the delamination length from the piano-hinge and \( l_1 \) (mm) is the vertical distance from the centre of the loading pin to the mid-plane of the specimen. The correction factor \( \Delta \) (mm), represents the absolute value of the delamination length, \( a \) when the compliance, \( C \) (mm/N), is zero. It can be seen from equations 5.1 and 5.2 that the fracture toughness correction factor can be ignored for small values of \( \frac{\delta}{a} \).

5.4 Result and Discussion

The results of the ‘initial’ and the ‘repair’ DCB test for both group are discussed below under the group’s name heading along with their fractograph analysis of the adherends’ failure surfaces.

5.4.1 Ambient

Initial & Repaired State

The results for both the initial and the repair stages are presented in Table 5.3. Specimen DB-3 experienced a hinge failure while testing after it had been repaired. The specimen’s contribution has been discounted and discussion has been limited to the remaining five specimens: DB-1, DB-2, DB-4, DB-5 and DB-6.
5.4 Result and Discussion

Also, the load-displacement plots for DB-1 and DB-4 at both stages of testing are shown in Figure 5.2 and Figure 5.3. These two figures used in this discussion represent two distinctive characteristics of the coupons, especially after repair. The illustrated figures show load rising quickly, almost linearly, in both cases for the pristine condition and the post repair. This can be attributed to the initial opening of the crack as initiated by the presence of the aluminium foil. A linear increase in load is observed until rupture of the bonded laminate occurs and the crack propagates through a mid-plane matrix (initial) or matrix-adhesive layer (post repair). In the case of the pristine samples, which had unadulterated glass fibre-polyester matrix interface, the load rarely rises past the initial threshold load at which the first matrix bond on the crack path was broken. Thereafter, the load drops steadily as the vertical displacement, $\delta$ and crack length, $a$ increase.

<table>
<thead>
<tr>
<th>Specimen DB</th>
<th>Fracture Toughness (N/m)</th>
<th>Max Force (N)</th>
<th>Strain Energy (Joules)</th>
<th>Fracture Toughness (N/m)</th>
<th>Max Force (N)</th>
<th>Strain Energy (Joules)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>356.31</td>
<td>30.26</td>
<td>1577.22</td>
<td>517.07</td>
<td>35.70</td>
<td>1627.62</td>
</tr>
<tr>
<td>2</td>
<td>397.92</td>
<td>30.57</td>
<td>1421.88</td>
<td>416.06</td>
<td>29.47</td>
<td>1493.53</td>
</tr>
<tr>
<td>3</td>
<td>390.99</td>
<td>24.73</td>
<td>1226.83</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>4</td>
<td>323.20</td>
<td>25.52</td>
<td>1027.18</td>
<td>392.50</td>
<td>29.90</td>
<td>1570.87</td>
</tr>
<tr>
<td>5</td>
<td>300.14</td>
<td>27.13</td>
<td>1285.34</td>
<td>357.19</td>
<td>27.44</td>
<td>1472.59</td>
</tr>
<tr>
<td>6</td>
<td>312.18</td>
<td>28.77</td>
<td>1457.55</td>
<td>438.32</td>
<td>32.80</td>
<td>1649.36</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th>Initial</th>
<th>Repaired</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Fracture Toughness</td>
<td>Max Force</td>
</tr>
<tr>
<td></td>
<td>(N/m)</td>
<td>(N)</td>
</tr>
<tr>
<td></td>
<td>Average</td>
<td>Standard Deviation</td>
</tr>
<tr>
<td></td>
<td>337.95</td>
<td>39.51</td>
</tr>
</tbody>
</table>

Table 5.3: Average fracture toughness: pre and post application of CA adhesive

As the Figures and Table 5.3 show, the introduction of CA adhesive at the fibre-matrix interface replicated similar behaviour of the initial specimens, especially
5. EXPERIMENTAL INVESTIGATION ON DELAMINATION-INTERLAMINAR FRACTURE

Figure 5.2: DB-1: Load-Displacement

Figure 5.3: DB-4: Load-Displacement
after the initial peak load. It appears that the CA bond was able to maintain a roughly average constant load even as the displacement and crack length increases. This means that the fibre-matrix interface had been slightly toughened by the adhesive. Therefore, slightly higher load is required to delaminate or fracture the repaired laminates beyond the first bond, with same crack length and vertical displacement as the initial specimens. Table 5.3 shows the average maximum delaminating load, which occurs at the initial bond breakage, increasing from approximately 28 N to 31 N for the initial and repaired specimens respectively. This represents a small improvement of approximately 9% in the resistive force to delamination. However, Figure 5.2 and Figure 5.3, as well as those in Appendix B¹, clearly demonstrate that the adhesive improves the resistive force to delamination as the crack grows.

Furthermore, it appears the stronger CA bond at longer crack length, a, compares to the initial fibre-polyester matrix bonding improves the fracture toughness of the repaired specimens as Table 5.3 shows. Upon repair, the coupons' resistant against fracture had improved by approximately 25%, from approximately 337 N/m to 424 N/m with standard deviations of 39.5 N/m and 60 N/m for the initial and repaired specimens respectively. These standard deviations are of similar ratios to their respective fracture toughness and therefore indicate the coupons exhibit similar behaviour and thus justify attention to detail during the repair process. In addition, to justify the superior toughness of the CA adhesive placed in-between the polyester matrix and glass fibre, the area under each load-displacement curve was computed as the strain energy. The initial DCB specimens had an average strain energy of approximately 1350(210) J, while the repaired had 1560(78) J. This translates to approximately 16% increase in strain energy above the initial value. This can be attributed to the higher delaminating load of the repaired samples above those of the initial specimens, beyond the initial peak load as the crack propagates.

¹Appendix B shows the remainder of the Load-Displacement plots and the R-Curves for other specimens
5. EXPERIMENTAL INVESTIGATION ON DELAMINATION-INTERLAMINAR FRACTURE

The 'repaired' plots in both Figure 5.2 and Figure 5.3 are characterised with sharp saw-toothed profiles in comparison to their corresponding 'initial' plots. This reflects a stick-slip behaviour at the cyanoacrylate-polyester interface. At each peak, the propagating crack is stopped briefly by the adhesive bond until sufficient load or energy is accumulated to rupture the cured adhesive molecular bond. The stored up strain energy is therefore adequate to cause a rapid rupturing crack propagation through the weakly bonded sections between the adherends' halves causing consecutive falls in the load magnitude, especially on specimens DB-4 and DB-5. These stick-slips marginally affected the average fracture toughness of the specimens, particularly DB-4 and DB-5. This fact is also reflected in the R-curve plots in Figure 5.4 and Figure 5.5 which represents the coupon's resistant to delamination or fracture at every measured crack length, \( a \). The R-curve usually shows quick rising of the fracture toughness with respect to the delamination length. It settles at a constant value with small variation as the propagation continues such that the lower load, \( P \) is complimented by higher displacement, \( \delta \) according to equation 5.1. The 'initial' R-curve of DB-4 shows more resemblance to the expected profile compares to the 'repaired' plot. The resulting change in the data distribution and the plot profile can be explained by superiority of the introduced adhesive compare to the initial polyester matrix bond. The general trend of the 'repaired' fracture resistance curves demonstrates the uniformity in the adhesive concentration, by volume, on the bonded plane.

Fractographic Analysis of the Adherends' Surfaces

Post Initial DCB Test

The failure surfaces after the initial DCB test had been performed on the specimens is shown in Figure 5.6. Those of DB-5 and DB-6 also show similar failure pattern. The delaminated surfaces were digitally scanned using an optical scanner from which the histogram images used in all the fracture surface analyses were developed. The Figure 5.6 shows the edited histogram graphic of the fractured surfaces of the adherends. This enhances the quality of the surfaces by manually controlling the amount of light reflecting on them. In Figure 5.6 and subsequent histogram graphics, the glass fibres reflects more light, appear brighter, while the
5.4 Result and Discussion

Figure 5.4: R-Curve: Fracture resistant of DB-1

Figure 5.5: R-Curve: Fracture resistant of DB-4
polyester matrix absorbs light and therefore appears darker. The failure surfaces in the figure can be described as 'matrix failure' of similar analogy to the bond failure types discussed in Section 3.2.1, Figure 3.2a. One adherend appears to be clean of the resin matrix while the other has a larger proportion of the matrix still attached. This matrix debonding shows no traces of fibre-strands pulling or fibre bridging which would otherwise suggest slight adherend failures. In essence, the initial DCB test shows the bonding and toughness weaknesses of the polyester matrix.

Also, the configuration of the fibre strands, in a closely knitted woven-roven, prevents adequate bond formation from contacts between two adjacent matrix layers separated by a single fibre clothing. Although this can be attributed to the viscosity of polyester resin and also possible flaws associated with the fabrication process. Some of the flaws include: improper wetness of the fibres by the resin matrix; inadequate matrix applied to each layer, especially those at the failure plane; possible presence of oil, dirt, and other surfactants that enhance weak matrix adhesion to fibres.

*Post Repaired DCB Test*

Figure 5.7 shows the histogram graphic of the failure surfaces of the adherends after the repaired DCB test had been conducted. It can be seen that all specimens demonstrate similar trend of failure; therefore, specimen DB-4, shown in Figure 5.8, is used in analysing the post repaired fractured surfaces in comparison with the pre-repair surface condition. The illustrated figures and visual observation show the specimens exhibited inter-adherend failure (major), and less adhesive failure. The adherend 'A' of the 'pre-repair', Figure 5.8a, shows a surface almost clean of polyester matrix while the adjacent adherend 'B' is covered with the matrix. However, the post repair DCB in Figure 5.8b shows a transformation, majority of the matrix had adhered to the adherend 'A'. This failure is regarded as inter-adherend, since the failure occurred in between the initially bonded (from fabrication) polyester matrix and underlining glass fibre at the mid-plane. At the failure plane, the matrix and the fibre denote a single layered composite, which
Figure 5.6: Pre-repair (initial) delaminated surfaces of the specimens
forms the top layer of one of the adherends, while the other adherend’s top bonding surface had less matrix content. It can be said that the adhesive was able to develop stronger bonds with exposed glass fibre on one adherend and also with the polyester matrix on the other adherend. Therefore, the failure can further be explained as ‘intra-laminar’ between the CA adhesive and the matrix at the failure plane.

Also, there are few cases of adhesive failure, like the red circled area in Figure 5.8b. These areas, and other similar ones, retained their initial pre repair forms after the adhesive had been applied. This shows that the adhesive was not able to form proper adhesion between the glass fibre and the polyester matrix.

The inter-adherend failure explains the similarity and the resemblance in the load-displacement behaviour (in terms of ductility, and even similar but slightly higher load) of the specimens for the ‘initial’ DCB and the ‘repaired’ DCB tests. Similar test of this nature has been reported to have been characterised with stick-slip, (Bader et al., 2000). However, such occurrence was minimised or rather suppressed because of the inter-adherend failure. It created a model of the initial DCB test where the fracture toughness is measured between the primary constituting materials, the glass fibre and polyester matrix. Furthermore, the results presented in Table 5.3 does not reflect or justify the bonding strength of the CA adhesive used. It can be said that the propagating crack, $a$, was diverted to the least resistance path in the case of the repaired samples. In this case, the glass fibre-polyester matrix section represents the weaker bond section near the test mid-plane. This shows the adhesive toughness in resisting delamination and also calls for careful preparation of the materials at fabrication stage.
Figure 5.7: Post-repair delaminated surfaces of the specimens
5. EXPERIMENTAL INVESTIGATION ON DELAMINATION-INTERLAMINAR FRACTURE

5.4.2 Simulated Environmental Impact

The discussion below is divided into two parts: the first part outlines the initial DCB test results, which quantify the initial fracture toughness of the polyester/glass fibre composite coupons. The second part reports the results for the repaired coupons where the CA adhesive replaces the polyester bond at the mid-plane. An 'r' subscript was added to the nomenclature to identify the results for the repaired specimens.
Initial DCB Tests: Polyester Mid-plane Bond Evaluation

The four groups of cast coupons were subjected to the first DCB test allowing statistical analysis of the fracture load and toughness of the prepared composite. Figure 5.9 shows the load against displacement curve for a representative specimen from the control group. An x-axis offset has been introduced for clarity. The linear phase is attributable to the separation of the adherends due to the presence of the aluminium foil. The load increases until rupture of the bonded laminate occurs at the point where the pre-crack aluminium-insert ends. The maximum load was observed just before the crack initiated from the end of the aluminium foil insert. This initial peak load was averaged for each group and the values are presented in Table 5.4 (third column). A drop in load reflects a release of energy as the crack propagates through a mid-plane matrix layer. For the case of a uniformly bonded coupon, the load/displacement curve would be expected to level out or taper off after fracture (Bader et al., 2000) indicating a uniform resistance to delamination as the crack propagates. However, the results presented here exhibit some low amplitude peaks and troughs in the load/displacement curve as a direct result of small defects observed on the matrix layer of the delaminated plane. These defects decrease the matrix/fibre concentration and lead to some minor stick-slip behaviour such as that reported for E-Glass/Epoxy composites (Bader et al., 2000; Kessler & White, 2001). During the DCB test, all specimens exhibited a propagating crack with small deviation from the mid-plane. There was no sudden change of the crack orientation or angle. Figure 5.10 shows the compliance cube root as a function of delamination length for specimen Ctrl$_1$, one of the designated control specimens. The $G_{ic}$ values are reported in Table 5.4.

Post-repair: CA Mid-plane Bond Evaluation

The post-repair DCB results from the control group (Ctrl$_{1-4}$) were analysed to determine the performance of CA as a repair agent under normal temperature and pressure. It was found that the repaired specimens exhibited an average initial peak load 29% higher than that observed for the pre-repair (first DCB) test results (Table 5.4). This can be attributed to the possibilities of stronger bonds formed
between the adherends through the CA adhesive and small opened pockets or voids along the delaminated plane being filled the adhesive. The former indicates that the CA based repair has higher bond strength than the cast interlaminar polyester layer.

A load/displacement plot showing the pre and post repair data for a representative specimen of a given group are shown in Figures 5.11 and 5.12, 5.13 and 5.14. For all groups the post-repair data (CA bonded specimens) are significantly more saw-toothed in nature than the curves shown in Figure 5.9 for the original specimens. The large amplitude peaks and troughs are characteristic of unstable crack propagation at the CA/composite interface, the stick-slip behaviour (Bader et al., 2000; Bardis & Kedward, 2001). This is due to the brittle nature of CA adhesive.
5.4 Result and Discussion

<table>
<thead>
<tr>
<th>Group</th>
<th>Condition</th>
<th>Load, P(N)</th>
<th>Fracture toughness, (G_{lc}) (N/m)</th>
<th>Toughness, (G_{lc}) (N/m)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Pre Repair</td>
<td>*Post Repair</td>
<td>Change in (P_{pk}) (%)</td>
</tr>
<tr>
<td>Ctrl(<em>1) (</em>{-4})</td>
<td>20 °C</td>
<td>49.61 (14.09)</td>
<td>64.87 (28.22)</td>
<td>30.76</td>
</tr>
<tr>
<td>Cold(<em>1) (</em>{-4})</td>
<td>-20 °C</td>
<td>47.02 (3.12)</td>
<td>43.89 (6.92)</td>
<td>-6.65</td>
</tr>
<tr>
<td>NS(<em>1) (</em>{-4})</td>
<td>40 °C, 98% RH</td>
<td>47.97 (5.59)</td>
<td>62.46 (15.65)</td>
<td>30.21</td>
</tr>
<tr>
<td>S(<em>1) (</em>{-4})</td>
<td>35 °C, 100% RH</td>
<td>55.56 (12.34)</td>
<td>80.10 (18.17)</td>
<td>44.17</td>
</tr>
</tbody>
</table>

* Denotes post-exposure (different environmental conditions) DCB test results. The standard deviations are quoted in brackets.

Table 5.4: Average peak load per group and the corresponding fracture toughness and not the environmental conditioning as all groups, including the control group which was stored under ambient conditions after repair, exhibited similar DCB results.

For each group the standard deviation of the initial peak load values has been calculated and presented in Table 5.4 underneath the average initial peak load value. All the groups showed improved results in comparison with their corresponding 'pre-repair' data apart from the group exposed to minus twenty degree Celsius (-20 °C). The average of the initial peak load for the repaired samples showed an increase of 30% and 44% for 'NS' and 'S' groups, respectively. Comparably, Table 5.4 and Figure 5.15 illustrate that increases of 67%, and over 100% in toughness to delamination initiation were achieved by the 'NS', and 'S' groups respectively portraying the success of the CA bonds in repairing delamination. The excellent performance of the samples at 35 °C exposed to 5% salt spray saline environment suggests that the combination of temperature and salt solution enhanced the cur-
5. EXPERIMENTAL INVESTIGATION ON DELAMINATION-INTERLAMINAR FRACTURE

Figure 5.10: A typical compliance cube root plot

ing of the adhesive which is a positive outcome for marine applications. It is not purely attributable to the temperature as the samples with 0% salt spray at 40°C did not achieve the same level of increase in performance. In contrast to this, the specimens subject to cold conditions exhibited a 6% drop in the group’s initial average peak load and had the lowest recorded fracture toughness of approximately 115 N/m. Although this represents over 30% loss in toughness, it still can be classed as a successful repair given the recovery from total delamination. It does highlight a durability issue in cold conditions which requires further investigation. Overall, the results demonstrate the durability and suitability of the CA adhesive for repair of polyester based composites that are exposed to severe environmental conditions.

Post-repair: $G_{Ic}$ Analysis and Stick Slip Behaviour

The unstable crack propagation exhibited by the repaired specimens complicates the fracture toughness analysis. The stick-slip behaviour significantly reduced the
5.4 Result and Discussion

![Graph showing bond strength](image)

Figure 5.11: Pre and Post CA adhesive repair bond strength of control specimen (Ctrl₂ and Ctrl₂r)

Data points available for the evaluation of the average fracture toughness of each specimen. At each peak, the propagating crack is stopped briefly by the CA bond until sufficient load is accumulated to cause an intralaminar failure, mostly. The stored up strain energy is therefore sufficient to cause rapid crack propagation resulting in an immediate fall in load. As a result, tests on 7 coupons quickly terminated in total delamination. A number of factors contribute to the variability in the bond strength: (i) degree of curing of the adhesive, (ii) The brittleness of the adhesive in comparison with the ductility of the initial polyester bond, (iii) The presence of of voids at the mid plane due to imperfect mating of the adherends at the repair stage which were subsequently filled with CA adhesive. Given that all repaired specimens had a minimum curing time of 1000 hours before DCB
5. EXPERIMENTAL INVESTIGATION ON DELAMINATION-INTERLAMINAR FRACTURE

Figure 5.12: Comparison of the polyester bond specimen (Cold2) with the CA bond specimen (Cold2r)

testing, the issue of uncured adhesive can be discounted. The brittleness of CA adhesive is a characteristic property and despite the use of a rubber toughened product here it does account for the stick slip behaviour observed. However, the primary source of variability in the results in Table 5.4, has been identified as imperfect mating of the adherends and the presence of voids or adhesive filled cavities. In the case of voids, delamination will be accelerated while in the case of adhesive filled cavities the delamination propagation can be impeded in a similar mechanism to that reported by Norris et al. (2011) for repair of GRP composites.

This initial toughness reflects the amount of energy required to breech the interlaminar bond between the adherends. Consequently, the fracture toughness ($G_{ic}$) values reported here were evaluated at the first peak load on the load-displacement curve, which denotes the onset of delamination. The initial $G_{ic}$ is based on the corrected beam theory, equation A.8, and setting the correction factor, $\Delta$, to zero at the reference point for the $G_{ic}$ calculation. This approach was used as it has
been shown that unstable variation of the value for $\Delta$ (sometimes also leading to unexpected low values of the toughness not reflecting the actual material behaviour) can be discounted and replaced with zero value (Brunner et al., 2006).

The $G_{ic}$ values calculated from the initial DCB tests exhibit an acceptable standard deviation presented along with the results in Table 5.4. The values for the repaired specimens exhibit a much higher standard deviation demonstrating the difficulty in calculating accurate fracture toughness for specimens that exhibit stick slip behaviour (Bader et al., 2000). Despite the variability in the $G_{ic}$ values, they are useful to show that the fracture toughness of the repaired specimens, including those exposed to harsh environmental conditions, is generally no worse than that of the cast specimens and in some cases can be higher. Therefore, if the voids between the bonded surfaces of the adherends could be reduced through better clamping/alignment, a more consistent bond strength could be achieved resulting in a more consistent $G_{ic}$ which would be expected to be in line with the higher values observed here. Introduction of the repair agent immediately upon onset of damage rather than after total delamination should result in superior mating of the surfaces and consequently a more effective repair. The average

Figure 5.13: Comparison of polyester bond and the environmentally treated CA adhesive bond (No Salt)
5. EXPERIMENTAL INVESTIGATION ON DELAMINATION-INTERLAMINAR FRACTURE

Fractographic Analysis of the Adherends Surfaces

The cast specimens of the environmental simulated impact were examined after the initial DCB test. It was found that they exhibited exposed glass fibres at the mid-plane indicating inter-adherend failure (Section 3.2.1), as expected. It should be noted that in the repaired specimens the Loctite 435 adhesive layer had bonded to the matrix and upon delamination, glass fibres were exposed.
After repairing and re-testing, visual examination identified freshly exposed glass fibre strands and confirmed inter-adherend failure. This failure type and the exposure of glass fibres are further evidences that a strong Loctite 435 bond was achieved verifying the suitability of CA adhesive as a repair agent for fibre reinforced polyester composite materials.

The delaminated surfaces were digitally scanned using an optical scanner. Figures 5.16, 5.17, and 5.18 illustrate the type of bond failure that occurred for most of the specimens. Cohesive failure was identified only in a few cases. Most of the cases however, led to adhesive failure. For the latter, the adhesive was observed to have been bonded to one half and left a trace of peeling on the other half. The sandwich nature of the repaired specimen with composite-adhesive-composite structure implies that at any given cross section, there are two interfaces, and the weaker of these debonds upon delamination. The image clearly shows zones of adhesive failure that alternate from one adherend to the other depending on which interface is the weaker. This accounts for the erratic manner of the delamination propagation, which was a chronic attribute of the CA bonded specimens in contrast to the smoother, continuous delamination of the uniform polyester layer in
the pre-repair composite. The optical images also revealed the residual texture of the cured adhesive on the delaminated adherends' surfaces. Analyses of the image of the delaminated halves of specimen Cold$_5$, Figure 5.16, showed that a large proportion of the surface area has the evidence of cohesive failure due to smoothness of the Loctite 435 on each half. The specimen had a low maximum delaminating load of 50 N. However, it showed more ductility as compared to some other specimens such as for Cold$_2$, Figure 5.17. Specimen Cold$_2$ was more brittle and was characterized by stick-slip each time the propagating crack extends beyond the well bonded regions. Figure 5.18 relating to a control specimen indicates that these regions show up as bright areas as the exposed glass fibres reflect light. Visual observations revealed loose glass fibres. This clearly demon-
strates that those regions had an inter-adherend failure. Approximately 80N was recorded as the maximum delamination load for Cold2r specimen.

It appears that the transversely oriented fibres, acting as cross binders to the major reinforcing longitudinal unidirectional fibres, were exposed because of inter-adherend bond failure. Furthermore, they acted as the major restraint on the delamination propagation path as shown in Figure 5.17. This behaviour was also repeatedly observed in most of the specimens which experienced the stick-slip and had higher delamination initiation loads, e.g specimen Ctrl1r. In Figure 5.18a, the specimen displayed major stick-slip at approximately 20 mm beyond the aluminium foil which corresponds to the location of the first adhesive bond failure of the bonded mid-plane. A stick-slip of 35 mm was recorded with corresponding loads of 83 N and 8 N for the initiation and the propagation-arrest crack lengths, respectively. The severity of the adhesive failure was impacted on the other substrate’s half with an apparent imprint. Also, it can be said that the propagation went through the path of least resistance, the weakly bonded regions. High density of the weakly bonded sections in proximity of those with adhesive bond failure potentials, adversely affected the overall resistive force of the specimens to delamination.

5.4.3 Ambient and Environmental Impact

The two groups showed different results and exhibited different characteristics. First, the environmental samples showed much higher initial delamination load at both stages of the DCB test and shorter loading pins’ displacement. This can be attributed to the type of glass fibre used, the unidirectional fibres. The fibre size and the space between consecutive strands promotes better adhesion between adjacent layers which was inadequate in the woven roving used in the other test. This was evidence in the observed loose fibre strands during initial DCB test for the environmental samples. Furthermore, the larger cross-sectional area of the unidirectional fibres enhanced stronger bond formation with the CA adhesive upon repair as the ambient specimens have shown that the adhesive adhere strongly to glass fibres.
Figure 5.18: a) Histogram image of Ctrl_{ir}, b) Load displacement plot for Ctrl_{ir}.
Second, there were disparities in the behaviour of the specimens upon repair. The environmental specimens were hampered with stick-slips which had been discussed in the preceding sections. The ambient specimens on the other hand displayed similar ductility of the pre-repair (initial) DCB test. These differences can be pinned on the associated flaws of the repair process. Also, the length of time involved between the repair and the second DCB test appears to have influenced the load-displacement behaviour. For the ambient (ductility), second DCB was performed after twenty-four hours, while it was over one thousand hours for the environmental class (stick-slip). This shows the adhesive continues to cure even after reaching considerable strength over a short period of time. This also means that areas of improper register between the two adherends loose their bonding to the adhesive as curing continues. This explains the areas with cohesive failures on the repaired environmental specimens.
5. EXPERIMENTAL INVESTIGATION ON DELAMINATION-INTERLAMINAR FRACTURE
Chapter 6

Self Repair Technique - The Healing Response

6.1 Introduction

The chemistry of cyanoacrylate adhesive as a healing agent has been discussed in Chapter 3. The robustness of the repairing system with cyanoacrylate adhesives has been evaluated using DCB tests, and the results from the simulated extreme environmental conditions have also been presented in Chapter 5. The use of hollow glass fibre (HGF) in vascular method of self healing has been met with some measures of success and drawbacks such as: adhesive impediment, hollowness reduction, cost etc. This chapter reports on the design and application of a vascular network without the use of HGFs in an FRP structure. It starts with a design concept for the non-HGF usage, followed by a flexural test experiment on a set of bulky glass-polyester composites. The chapter concludes with a similar test on slender composites with hollow network diameter of 400 μm.
6. SELF REPAIR TECHNIQUE - THE HEALING RESPONSE

6.2 Non-Hollow Glass Fibre Based Self Healing Method

An HGF inspired self healing method is considered in this thesis for active repair or mitigation of cracks in FRP structures such as WTB. As discussed in Chapter 2, HGF has widely accepted advantages over the micro-encapsulation method. Among the host of benefits include: increased storage facility of the healing agents and ease of incorporation of the system into the fabrics of the material’s architecture. The possibility of exosystemic storage is postulated to enable the sustainability of the system and also to enhance the physical or chemical manipulation of the healing agent(s) or changing the fluid to suit the latest threat. It was stated in Section 2.5.2 that the latest generation of self healing systems, the 3D interconnected HGFs, are still at the conceptualisation stage. The challenge remains the incorporation of the 3D edifice into FRP composite architecture without dislodging the reinforcing fibres.

However, among the first generation self healing works, those in 2D have been widely reported (Bleay et al., 2001; Motuku et al., 1999; Pang & Bond, 2005a,b; Trask & Bond, 2006; Williams et al., 2008a). A report by Bleay et al. (2001) on HGFs was widely accepted and many had based their studies on this finding that HGFs are not mechanically detrimental to the functionality of their host. However, they make no significant contribution towards the host's strength. 2D or 3D HGFs pose different challenges of their own. A particular threshold of energy is required to cause a penetrating crack through the hollow fibres before healing can be activated. Otherwise, damages would go unrepaired in case of a fracture event if the healing agent’s containing vessel, the HGFs, cannot be fractured due to insufficient energy which might have been dissipated before reaching the HGFs. Another drawback is the impediment on the crack path by HGFs’ debris, hindering the seepage of the repairing agent from the fractured HGFs. In this scenario, viscous healing fluid would have to navigate between two imposing walls, namely the HGF and the immediate laminate walls enroute to the crack path. Repairing
6.2 Non-Hollow Glass Fibre Based Self Healing Method

agent had been reported to have cured inside its embedded fractured HGF container, exhibiting disparity in mixing with the hardener, a prerequisite condition for self healing (Pang & Bond, 2005a; Williams et al., 2008a).

This chapter addresses the issue of HGF sabotaging its most essential functionality in a self healing system. It proposes a more economical 2D model of an alternative route for delivering the repairing agent to its needed location without engaging the use of HGFs. This involves creation of hollow network passages within the material architecture at the fabrication stage. Laminate constituting matrix forms the walls of a series of hollow sections same as the walls formed from ink writing-scaffolding, and rapid electric discharge for 3D network formation, (Huang et al., 2009; Lewis, 2006). The hollowness can be created using temporary formwork of removable material, which can be taken off from the finished product either through a chemical reaction process or by any easy mechanical means with minimum damage initiated in the material.

The vacuum left behind by the formwork acts as a delivery passage for hydraulic bolstered repairing agent into any damaged site within the material. This is similar to the use of HGF to store and deliver repairing agent to the required location. The system described here, requires no on-board storage of healing chemical, rather the healing agent is pressurised through the matrix walls from a supply reservoir. This creates a network of healing agent supply channels, eliminating undue obstruction of the adhesive from reaching the crack surfaces. Also, it creates more repairing avenues for cracks, which would otherwise have insufficient threshold energy to fracture an HGF, thereby increasing the recovered efficiency. In effect, depending on the viscosity of the repairing agent, every crack penetration path breaching the hollow walls acts as a seeping channel. The following experiments describe a test carried out to validate the outlined concept involving fabrication of a bulky, rectangular glass fibre reinforced polyester (GFRP) laminate thick enough to accommodate 3 mm holes drilled along its length and intersecting holes at a prescribed off-set. This concept is followed by three-point bending test on slender similar 2D model.
6.3 Specimen Fabrication and Test Preparation

Glass fibre polyester composite specimens were made following the manufacturer’s instructions. A polyester matrix was made using the same mixture of Norsodyne H 13212 TAE orthophthalic polyester resin with Metox-50, an MEKP curing agent. A unidirectional E-glass fibre (GRS), weighing 690 g/m², was used as the reinforcement. The matrix was quantified on supplier’s instruction at a ratio of 2.5:1 by weight of polyester resin to that of the glass fibre. A specially fabricated aluminium mould, dimension 300 mm long x 300 mm wide x 25 mm depth was used in forming the laminated composite. Twenty two layers of the glass fibre matings were cut to fit the mould’s dimensions for the fabrication of a cross-ply laminates of [0/90]_{11}. The manufacturing process followed the same procedure outlined for fabricated GFRP coupons in Chapter 5.

The laminated panel, while still in the mould, was allowed to cure at room temperature for 24 hours after which it was removed from the mould. It was cured for a further 24 hours at room temperature. The panel was then cut into five smaller coupons with dimensions of 150 mm long x 46 mm wide x 23 mm thickness, using a 5 mm mitre saw. The specimens were labelled alphabetically from A to E. Each specimen had two 3 mm diameter holes drilled on either side of the length and the width. The holes were centred at a depth of 7 mm below the mid-plane, and with an offset of 20 mm and 50 mm from the centre line along the width and length respectively, Figure 6.1. This creates a four-point intersection grid within each specimen. The grid was placed in this location due to higher tensile stresses, which the lower fibre layers experience under a bending flexural action. Likewise, it is postulated that since the matrix will yield earlier than the glass fibres under tension, cracks arising from such a damage would provide a leeway for the repairing fluid to reach other subsurface defects.

There were some difficulties in aligning the holes and in keeping the drilling bits from diverging from the main course, especially on the longer sections. To reduce the chances of divergence, some specimens were drilled from both sides to meet at the centre. Due to the proximity of the drilling to the outer surface and the
6.4 Mechanical Tests

Figure 6.1: Layout of the hollow network created into the specimens

material density, the drill bit came out at the wrong end, on the bottom surface of one of the specimens, \( E \). The affected specimen was subsequently withdrawn from the test. Afterwards, all the drilling inlet holes, except two (as indicated in Figure 6.1), were blocked to a depth of 2 mm from the surface using a two-part epoxy adhesive. Those left unsealed, one on either end of the length, were to serve as the healing agent injecting points as indicated in Figure 6.1. Cyanoacrylate (CA) based adhesive, Loctite 435 (Section 3.3), was chosen as the healing agent.

6.4 Mechanical Tests

The results reported here were obtained from a three-point bending test. BS EN ISO 14125 (BSI, 2011) was consulted as a guide for the testing procedure. The standard describes the procedure for obtaining the flexural properties of an FRP composite from a bending test. The structural properties such as the bending stiffness, \( EI \), at each developmental stage were obtained from the test. Three-point bending test was chosen damage initiator as oppose to indentation by impact damage as reported in some literature (Patel et al., 2010; Trask & Bond, 2006), and also to have consistency in damage initiation and assessment of the repair.
strategy. This enables close referencing of the recovered efficiency to its initial undamaged state assessed under the same testing regime. The specimens were simply supported on a 100 mm support span. The test was conducted in displacement control mode at a rate of 1 mm/min on a 100 kN load cell using Zwick Roell 1474 mechanical testing machine. The load was applied at mid-span through a cross bar across the width of the specimen. The load and central deflection data were automatically acquired by a computer connected to the testing device.

The necessary operating parameters such as the threshold load and the pre-loading speed were set to 30 kN and 8 mm/min respectively. The test was set to alter at 30% load drop off the peak load while the load is held on the material after damage had been initiated. This simulates a damaged state, which might be experienced in practice. The test lower termination value was to allow the development of fine cracks within 0.5-2 mm width. Wider crack beyond this range would render the CA adhesive ineffective as it lacks gap filling properties. Developing cracks were monitored and the width measured as they grow. This ties in with the goal of early prevention of fine macrocrack from propagating or increasing in width, and thus ensuring its development into delamination is not nurtured.

Analysis of Data

The effective stiffness was calculated using equation 6.1 with reference to the yield load and deflection. The equation is based on maximum deflection of a simply supported beam under a point load. When it is rearranged, it gives equation 6.2, the effective stiffness, \( EI \). Where \( \frac{\Delta P}{\Delta \delta} \) is the slope of the linear portion of the load-deflection plot, and \( L \) is the span length between the supports. The results were evaluated on the specimens' stiffness, \( EI \), rather than the conventional load carrying capacity in order to account for the slight variations in the dimension of the specimens.

\[
\delta = \frac{PL^3}{48EI_{eff}}
\]  

(6.1)
6.4 Mechanical Tests

\[ EI_{\text{eff}} = \left( \frac{\Delta P}{\Delta \delta} \right) \frac{L^3}{48} \]  

(6.2)

\[ \text{Efficiency, } \eta = \frac{\text{Post repair } EI_{\text{eff}}}{\text{Pre repair } EI_{\text{eff}}} \times 100\% \]  

(6.3)

The slopes were calculated using the secant modulus method. The secant modulus is usually used in analysing the non-linear behaviour of polymer plastics. It is computed between two data points, between the origin and the point of interest on the stress-strain or load-deflection curve. Within the linear range or below the yield limit of a load-deflection curve, the secant modulus is equivalent to Young’s modulus. An example is shown in Figure 6.2 for fibre glass reinforced polymer composite.

Figure 6.2: An example of secant modulus within the linear range
6. SELF REPAIR TECHNIQUE - THE HEALING RESPONSE

6.4.1 Self Healing Procedure

At the test termination point, while the load was being held constant on the specimen, a quantified volume of Loctite 435, the healing agent, was released under pressure into the hollow network via a hand held syringe. The CA adhesive was pumped into the specimens from both sides of the injection points. The specimens were then (turned over on their sides and) allowed to cure under ambient conditions for 36 hours. The repaired specimens were tested under a three point bending flexural test to compare the material's effective stiffness with those obtained from the first test. The efficiency of each repaired specimen was calculated from the comparison according to equation 6.3. Each specimen was analysed on its own merits due to the non-generic flaws in their physical characteristics. Specimens (C and the discounted E) that were cut-out from an area closer to the edges of the parent laminate were observed with curved fibres at their edges. Voids volumes were left at these places between adjacent plies. These make the specimens characteristically different from those cut out from the laminate's centre. Since the production process was not mechanically automated, it was expected that certain features such as imbalanced resin volume between the layers would become an inevitable flaw.

6.5 Results and Discussion

Pre-repair

Control Test

Before the specimens were drilled, they were first subjected to a three point bending test as outlined above. This was to enable a comparative study of the all results. The specimens were loaded between 0-13 kN to determine the initial mechanical behaviour in the absence of the drilled holes. This load range was chosen after a dry test run on a similar sample. Afterwards, it was adjudged that the load range will not be detrimental and would allow proper load-deflection profile to be developed. The result of this preliminary exercise is provided along with the subsequent pre-repair and post-repair test following the drilling.
6.5 Results and Discussion

Pre-repair

The load-deflection curves for each specimen show the specimen's behaviour before drilling, after drilling (pre-repair) and post-repair. These are presented in Figures 6.3, 6.4, 6.5 and 6.6. It can be seen that the 'Undrilled' and the 'Pre-repair' curves follow similar paths with reasonable variation from the average slope path. This shows that the anomalies and discrepancies from the fabrication method had little effect on the specimens' mechanical capacities. Aside from Figure 6.5, these plots show that the drilling had less detrimental effect on the laminates' mechanical capabilities. On the flexural stiffness, a reduction of approximately 7%, 1%, 17% and 2% were noted for specimens A, B, C and D respectively. The high loss of stiffness in specimen C after drilling can be ascribed to inflicted damages from the drilling action. The mechanical evaluation of the specimens' performances from the drilling to infliction of damages are numerically illustrated in Table 6.1. Due to the non-linear load-deflection behaviour, often synonymous with polymeric material, secant modulus was used in computing stiffness values presented in Table 6.1 according to equation 6.2. The secant slope was calculated between the origin and the data point at 12 kN, judged to be within the linear phase than the non-linear. The sudden load drop in the load-deflection plots denotes the end of the test. It appears that the drilled (pre-repair) specimens experienced some progressive minor damages immediately beyond their respective yield points, as shown by the pattern of the curves fluctuations, from smoothness to slight roughness, characterising sudden intermediate load drops. This occurrence can be attributed to the damage initiated by the drilled holes, the rapidly increasing matrix cracking, and unsustainable level of delamination eclipsing the specimens' bearing capacities.

The typical characteristic of FRP composite, exhibiting little or no plastic deformation before failure, was well portrayed by these specimens as shown in their load-deflections figures. It appears that the drilled holes had created weakened sections around their locations. Cracks formed were attracted towards the hollow networks as a result of the applied load being transferred to the supports via the
6. SELF REPAIR TECHNIQUE - THE HEALING RESPONSE

Figure 6.3: Specimen A: Pre & Post repair load-deformation plots

Figure 6.4: Specimen B: Pre & Post repair load-deformation plots
6.5 Results and Discussion

Figure 6.5: Specimen C: Pre & Post repair load-deformation plots

Figure 6.6: Specimen D: Pre & Post repair load-deformation plots
### 6. SELF REPAIR TECHNIQUE - THE HEALING RESPONSE

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Stage</th>
<th>$P_{yield}$ (kN)</th>
<th>$\delta_{yield}$ (mm)</th>
<th>FS (N/mm)</th>
<th>EI (Nm²)</th>
<th>$\eta$ of EI (%) post/pre</th>
<th>$\eta$ of EI (%) post/undr</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Undrilled</td>
<td>-</td>
<td>-</td>
<td>7332.9</td>
<td>152.77</td>
<td>102.17</td>
<td>94.95</td>
</tr>
<tr>
<td></td>
<td>Pre-repair</td>
<td>18.32</td>
<td>3.15</td>
<td>6815</td>
<td>141.98</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Post-repair</td>
<td>14.53</td>
<td>2.12</td>
<td>6962.7</td>
<td>145.06</td>
<td></td>
<td></td>
</tr>
<tr>
<td>B</td>
<td>Undrilled</td>
<td>-</td>
<td>-</td>
<td>8594.3</td>
<td>179.05</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Pre-repair</td>
<td>22.94</td>
<td>3.39</td>
<td>8501.3</td>
<td>177.11</td>
<td>87.76</td>
<td>86.81</td>
</tr>
<tr>
<td></td>
<td>Post-repair</td>
<td>18.50</td>
<td>2.59</td>
<td>7460.8</td>
<td>155.43</td>
<td></td>
<td></td>
</tr>
<tr>
<td>C</td>
<td>Undrilled</td>
<td>-</td>
<td>-</td>
<td>7712.1</td>
<td>160.67</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Pre-repair</td>
<td>23.34</td>
<td>3.95</td>
<td>6423.3</td>
<td>133.82</td>
<td>74.36</td>
<td>61.94</td>
</tr>
<tr>
<td></td>
<td>Post-repair</td>
<td>19.67</td>
<td>3.60</td>
<td>4776.6</td>
<td>99.51</td>
<td></td>
<td></td>
</tr>
<tr>
<td>D</td>
<td>Undrilled</td>
<td>-</td>
<td>-</td>
<td>8963.7</td>
<td>186.74</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Pre-repair</td>
<td>22.11</td>
<td>3.54</td>
<td>8746.5</td>
<td>182.22</td>
<td>80.86</td>
<td>78.90</td>
</tr>
<tr>
<td></td>
<td>Post-repair</td>
<td>14.95</td>
<td>2.46</td>
<td>7072</td>
<td>147.33</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

FS: The secant flexural slope from each load-deflection plot.

Table 6.1: Pre & Post-repair results: showing the recovered stiffness and the corresponding efficiency of the repairing agent

least resistance route. For example, specimen A had a shear crack failure pattern at 45 degrees causing short delamination (in between left-hand support and the left-sided sealed inlet) as the crack propagates downward, and a major layer separation originating from the centre of the hole towards the support, Figure 6.7. The sudden drop in load, which instantaneously concluded the test, revealed multiple layer failures as shown by the shear crack. It shows that the layers failed at similar load magnitude. This arises as compounding load is being transferred from one failed layer and it shared by the adjacent ones. This failure mode favours the test such that propagating cracks will have direct access to embedded hollow network, which will indeed influence flow of repairing agent to the required site(s).
6.5 Results and Discussion

Figure 6.7: Edgewise traces of the crack path on specimen A. The red line highlights the failure path induced by the shear crack as it changes to the delamination

Post-repair

The load-deflection curves and the numerical representation of the mechanical properties of the repaired laminates have been comparatively presented earlier in Figure 6.3 to Figure 6.6 and in Table 6.1. A subsection of Table 6.1 shows the recovered efficiency, $\eta$, by individual specimen. The 'post-repair' plots follow similar trend as those of the 'pre-repair' plots shown in each figure. It is quite apparent from the drop in load magnitude that the loading capacities of the specimens had been compromised. The peak load dropped on average by approximately 22%. Despite the significant drop in load, the repairing agent performed adequately well. To an appreciable degree, the initial reaction between the fibres and the matrix in transferring mechanical stresses was restored. This is evident in the replicated behaviour of the initial specimens up to the yielding points as shown in Figure 6.8. Though the sample size is small for a statistical analysis, the plot, Figure 6.8, shows closely matching properties, especially for specimens B, C and D. This implies that the restored stiffness values are a close representation of the laminates' pristine stiffness values as Table 6.1 reveals. Also, the repaired specimens showed improved ductility after the yielding points beyond which most of the specimens (with the exception of A&D) show plastic deformation while the
load drops off at a steady rate. The cause of this is not certain. Indentation of the cross-head loading bar shows a high possibility of this cause. The bar appeared to have been dented which caused unequal load distribution, and rotation at the support.

Figure 6.8: Comparison of load-deformation slopes

Specimens $A$ and $B$ showed the highest recovered stiffness with efficiencies of approximately 95% and 87% respectively, relative to the undrilled stiffness of the laminates. Specimen $D$ is somewhat similar in its stiffness responses with 78% recovered. As earlier discussed, specimen $C$, with the lowest recovery of 61%, appeared to have been extensively damaged by the drilling action. In the absence of non-destructive equipments to probe and visually grade the internal damages, it can only be stipulated that the internal architecture of the specimen had been severely damaged from the drilling and further compounded by subsequent loading actions. All but one of the specimens attained approximately 80% of their corresponding initial yield load, while specimen $D$ was within 73%
of its 'pre-repair' loading capacity. Figure 6.9 shows a bar chart representation of the results, which directly compares the effective stiffness of the specimens at stages of 'pre-repair' and 'post-repair'. An overall average of 88% efficiency was recovered. There are prospective avenues of improving these results given that the repairing agent used in this case was not chemically structured or engineered for this purpose. A major condition for utmost efficiency performance of the CA adhesive is the utilization in mitigating very narrow cracks.

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**Figure 6.9: Comparison of the effective stiffness**

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### 6.6 Automation of Repairing System (ARS)

The novel aspect of the concept of autonomous healing is the adaptation of 'artificial' intelligence. The state of the art of this technique requires minimum extraneous or human intervention. This embraces wider field of structural health monitoring (SHM). The owners of 21st century structures demand their structures
to be of high performance, durable, sustainable, efficient and aesthetically pleasing. Typically, SHM uses algorithms based on non-destructive methods such as piezoceramic actuator patches (PAP) (Ghoshal et al., 2000; Sundaresan et al., 1999) and those discussed in the literature review (Section 2.4.1). The PAP attached to the structure of interest excites it and the vibration responses are acquired through other sensor or using a different PAP or via a Doppler vibrometer. These advanced techniques are information based without any counteractive measure initiation. This section provides a two in one method associated with self healing, an incarnate of smart material. A test sample is simulated to be under damaging force and an accompanying device automatically initiates the self repair strategy depending on the damage intensity. In the preceding section, ‘self healing’ was carried out through a manual injection of repairing adhesive. In this section, the procedure is self activated by setting up a threshold deflection to activate the delivery of the adhesive.

6.6.1 Design of Automation System

The automated system was developed on a LabVIEW System Design Software (National Instrument), Figures 6.10 and 6.11. Maximum deflection is measured at the midspan via a pre-calibrated potentiometer. The sensor feeds the data to the LabVIEW interface. The program’s built in boolean algorithms check continuously through a while loop and ensures that the received data are within the acceptable range. Once exceeded, sufficient electric current is released from a connected 12 V power supply unit to an AC motor (the actuator in this case) to facilitate the flowing of adhesive from its storage, a syringe, into the specimen.

6.6.2 Automated Self Healing Test

The inclusion of the automated repair system did not require major changes to be made to the initial test set-up. The failed specimens were re-drilled to clear the network of cured Loctite 435. One injection point was used instead of two as it was in the previous case. The remaining points were sealed. A polyvinyl chloride (PVC) plastic pipe, 1000 mm long, 3 mm inner diameter was used as
a delivery passage from the external reservoir (syringe) to the injection point on the specimen. The 5 ml syringe was firmly mounted on a steel stand that also housed the actuator. A 5 ml volume of Loctite 435 adhesive was manually drawn into the PVC pipe using the syringe. This ensures shorter distance travel by the adhesive before reaching the intended target when the actuator supplies the necessary pressure to transfer the adhesive to the specimen. The operation starts by entering a 2 mm deflection limit, which is built into the LabVIEW program. The programme automatically reads in the current deflection. When the threshold value is exceeded, the actuator becomes active and drives in the pressure handle of the syringe. Using this ARS technique, Loctite 435 was observed flowing out at the failed sites on the specimens.
6. SELF REPAIR TECHNIQUE - THE HEALING RESPONSE

Figure 6.11: Labview: The operational interface

6.7 Vascular Network of String Formwork

Section 6.4 demonstrated a conceptual validation of self-walled hollow network passages. The section described the absence of intermediaries such as HGFs which are of no significant contribution but rather a disservice to the system. However, a self healing system incorporated into a structure should not be at the expense of reduced stiffness as it was for the earlier tested specimens. The tests conducted to examine the stiffness at every stage from the bulky specimens without holes to their corresponding repaired states, showed an average reduction of 19% in the recovered stiffness. This can be considered as a significant reduction of laminates' mechanical capabilities given that the holes make up approximately 7% of the cross-sectional area. Also, it was thought that the drilling action had initiated some damages via the creation of series of short fibre sections within the composite. The mechanics of stress transfer in short fibre composite is quite different from the continuous fibre reinforced composites.

In an FRP composite, there is a fibre length for load transfer. In a continuous composite e.g a unidirectional laminate, stress effects at fibre ends are deemed negligible due to the fibre's length exceeding the required load transfer length.
Also, the maximum fibre stress, $\sigma_{\text{max}}$, is limited by the fibre volume fraction in the composite (Agarwal et al., 2006). Conversely, this is not strictly true for short fibre composites. The maximum stress in a fibre is given as

$$\sigma_{\text{max}} = \frac{\tau_y l}{r}$$

where $\tau_y$ is the shear stress on the fibre-matrix interface, $l$ is the load transfer length and $r$ is the fibre radius. Equation 6.4 shows the importance of having adequate fibre length, which ultimately controls the laminate mechanical properties. Beyond the load transfer length, $l$, the fibre supports lower stress than $\sigma_{\text{max}}$. This would explain why there was a reduction in the stiffness of the bulky laminate after drilling due to permanent alteration of the internal architecture of the composite. Also, this method of post-fabrication retrofitting to accommodate interconnected hollow channels raises some doubts concerning the self healing status in its entirety. Hence, this section redresses the post-fabrication modification approach by making the desirable hollow network a native feature of the host laminate. This can be achieved through the inclusion of a two-dimensional temporary formwork, configured to the appropriate designed flow pattern for the repairing agents, and embedded into the laminate at fabrication stage. It can be randomly or strategically placed (at fabrication stage) between adjacent laminate layers of interest. After the laminate had been constructed, the formwork is then removed through least resistant scheme to avert or minimise any structural damage to the laminate. This can be achieved either through dissolving a fugitive formwork in a chemical solvent or using removable strings.

In the experimental tests described in this section, laminates containing this micrometer diameter orifice are tested in flexure to determine their flexural stiffness. The test is divided into two sections both aimed at generating damages (like cracks) internally, externally or both, in order to optimise the use of the cyanoacrylate adhesive. The first section explores the prospect of developing these minute cracks through damage accumulation by repeated loading—stress...
Figure 6.12: Layout of the hollow network configurations
debilitation or fatigue under few cycles. The second uses load diminishing capac-
ity of the laminate under a one-off scenario to signal the detection of targeted
level of damage.

6.7.1 Fabrication of Specimen

The assembly process was similar to the earlier reported test on the bulky speci-
mens. A laminate of 220 mm square was used in this case. First, the temporary
formwork was constructed using a 400 $\mu$m diameter plastic string of suitable ten-
sile strength. The strings were attached to a rectangular wooden frame for four
different sets of 2-D configuration of the hollow network as shown in Figure 6.12.
The 2-D connections at the junctions were achieved by perpendicularly laying the
strings over each other. The strings were held in position by passing the single
ones through an orifice and the coupled strings were separated by passing them
around a 6 mm diameter timber separator. The strings were locked down using a
timber peg, which also controls the tension in the strings by applying a downward
pressure, Figure 6.13. The tension was monitored to ensure that physical contacts
were made for the 2-D connections at the appropriate intersections.

Figure 6.13: Layout of the hollow channel network and its incorporation

In the laminate formation process, since the objectives of this exercise was more
inclined on demonstration of recovered strength through effective 2-D delivery channels than on the actual effect of different forms of glass fibre or that of the fibres orientations’ influence on the recovery, a different glass fibre fabric was used. Woven glass fibre of 200 g/m² was used instead of the initial unidirectional glass fibres. Ten layers of the glass fibre material measuring a total of 96.8 g were used. The fibre clothing for each layer was cut to the mould’s dimension of 220 mm x 220 mm. Unsaturated Norsodyne H 13212 TAE polyester resin of 181 g was portioned out at the ratio of 1:1.9. This was to reduce the resin volume to bolster the occurrence of delamination which was not largely obvious or observable from the test in Section 6.4. The hardener, MEKP Metox-50 was used at 2% of the resin mass for the resin polymerisation. The fabrication was conducted sequentially from the first layer to the fifth by applying the resin, spreading it with a paint brush on the mould followed by a meticulous placement of one glass fibre mat. At this point, the string formwork, which had been previously coated with honey wax (de-moulding agent) to facilitate easy post-curing extraction, was placed on the fifth layer such that the wired arrangement sits at the laminate’s mid-plane, and the layering order was resumed up to the tenth layer.

The laminate was allowed to cure for 24 hours after which the supporting timber frame was removed and the plastic strings were carefully pulled out. Complete extraction of the strings was not achieved as some broke in the process and were stuck inside. The cause of this breakage has not been extensively investigated. The weakening of the string’s strength due to the matrix curing temperature on the plastic can be ignored because some were successfully removed. Yet, the contraction of the polyester matrix after curing can be attributed to the plastic strings snapping off. At this stage, the de-moulding agent had solidified around the strings, and variation of temperature across the whole volume of the laminate allows partial phase changes of the de-moulding agent, which permits some removal.

The laminate measured an average thickness of 3 mm after curing. Afterwards, the laminate was machined to smaller specimens of dimension 25 mm (width)
x 80 mm (length) and later grouped according to their respective hollow configurations. This smaller hollow diameter ideology gives a 3% reduction for the hollowness contribution to the cross-sectional area (for the most congested configuration, VN-1, Figure 6.12) and with 0.53% contribution to the specimen’s volume as opposed to 7.34% for the bulky specimens.

6.7.2 Mechanical Test and Repair Strategy

The three-point bending test was set-up as shown in Figure 6.14 similar to the setting used in Section 6.4. The same principles as the previous ones were adopted in quantifying the bending stiffness following the BS EN ISO 14125 (BSI, 2011) guidelines, and using equations 6.1 and 6.2. Some test parameters were either changed or readjusted to comply with the change in specimen size. The support span was reduced from 100 mm to 64 mm between the supports, and the load cell on the Zwick Roell 1474 testing machine was replaced with a 10 kN load cell. Similar to previous test, the Zwick Roell was operated in the displacement control mode for the test at rate of 1 mm/min, while all other parameters were left unchanged.

Due to the small thickness of the specimens, the deflection control for ARS activation was not utilised. The small thickness of the specimens gives the likely scenario of a concurrent failure of the layers due to each layer having similar curvature from the bending action. As a result, the load will quickly drop off as the stress level exceeds the laminate ultimate strength. The alternative approach to engaging ARS is to have a full “Zwick Roell - ARS” integrated systems. These unified components will function on the automatic test shutdown from the Zwick Roell. This will act as a passive feeder to activate the hydraulic action of the actuator, thereby pressurising the flow of the repairing adhesive into the specimen via the connected delivery tube. This preference was however not employed due to the closed system functioning of the Zwick Roell.
At test conclusion, the test specimen is withdrawn and manually repaired using a syringe to pressurise the repairing agent into the specimen. Due to the volumetric constrain of the hollow passage, particularly at the entry point, a lower viscosity CA adhesive, Loctite 406, was used instead of the Loctite 435. For similar reasons, a clinical syringe fitted with hypodermic needle was employed to ease off the pressure at the ejection point. The repaired specimens were cured for 24 hours and were retested under same testing procedure after the adhesive was adjudged to have fully polymerised within the crack enclosures in the specimens. The contributions of the specimens with disparities, such as irremovable strings as discussed earlier, were discounted as they were omitted from the outlined test.
6.7.3 Result and Discussion

Strategising Failure Scenario

Cyanoacrylate adhesives are not known for gap filling capabilities and hence are seldom used in the world of structural repair (Petrie, 2006). In order to utilize the bonding effectiveness of a CA adhesive, the adherends must be in close proximity to a thin film of the adhesive. This translates to creating sufficient microcracks before the hollow connections are flooded with repairing agent. The damage control procedure was re-strategized by infusing the repairing agent into the specimen considering cases corresponding to the following four different conditions:

1. a threshold of 4 mm mid-span deflection, specimen A1
2. 7 mm mid-span deflection, specimen A2
3. inducing fatigue damage for 9 cycles, peak load of 600 N, specimen A3
4. 40% drop in the load carrying capability, specimen A4. A large margin of percentage drop in load was chosen to offset load fluctuations in the early part of the test at lower loads.

The specimens, labelled A1, A2, A3, and A4 (all of VN-2 configuration), were retested after curing period was over. The preliminary results showed that A1 and A2 had developed no structural damage at their time of repair. The plot of load-deflection of the repaired specimens depict the profile of that of a stiffer specimens than the virgin ones. However, this slightly improved stiffness can be said to have originated from the hollow connection passages being filled with the repairing agent. In this case both the matrix and the reinforcing glass fibres were still within their elastic limit. Specimens A1 and A2 showed similar responses to A3. From equation 6.2, the slope can be regarded as the prime term controlling the stiffness such that higher slope means stiffer material and vice versa. It can be seen, Table 6.2 shows close values of the slope plot for specimen A3. The slopes were obtained using the secant method (between 0-500 N), for each run of the fatigue test for specimen A3, including the repaired runs. The table shows
no indication of damage(s) in the pre-repair runs hence the similar post-repair stiffness. Also, a standard deviation value of 1.32 for the all the slope data in Table 6.2 confirms that the A3 is almost of the same mechanical strength as the pristine state. In this instance, the adhesive can be said to have shown filling capacity rather than healing competency in the absence of apparent damage.

<table>
<thead>
<tr>
<th>Test run</th>
<th>Slope (N/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>159.40</td>
</tr>
<tr>
<td>2</td>
<td>160.65</td>
</tr>
<tr>
<td>3</td>
<td>163.21</td>
</tr>
<tr>
<td>4</td>
<td>160.94</td>
</tr>
<tr>
<td>5</td>
<td>160.38</td>
</tr>
<tr>
<td>6</td>
<td>163.42</td>
</tr>
<tr>
<td>7</td>
<td>161.30</td>
</tr>
<tr>
<td>8</td>
<td>162.84</td>
</tr>
<tr>
<td>9</td>
<td>162.66</td>
</tr>
<tr>
<td>Repair 1</td>
<td>160.99</td>
</tr>
<tr>
<td>Repair 2</td>
<td>162.51</td>
</tr>
</tbody>
</table>

Table 6.2: Stiffness of A3 in terms of slope for 11 fatigue test runs. “Repair 1” and “Repair 2” represent the first and second post repair stiffness test runs.

The fourth criterion offered a viable chance of repairing real damages. However, specimens run a potential risk of more than the intended level of damage(s) being initiated. Macorcracks can rapidly grow and propagate to larger cracks due to the non-ductile nature of the specimens when the load begins to drop. The load-deflection plots, Figure 6.15, for both the pre and post repair A4 show similar profile up to 600 N. These parallel responses demonstrate that considerable amount of stiffness had been recovered. Quantifying the recovery relative to the maximum loads of approximately 945 N and 580 N for pre and post-repair A4 respectively, represent an efficiency of approximately 61% of recovered loading capacity. Table 6.3, shows the evaluation of the recovered efficiency from the stiffness perspective, using the secant modulus method, along the linear portion of both plots in Figure 6.15, in between 0-500 N. This shows that the efficiency of the repaired specimen is approximately 91% of its pristine state. From the analyses of the outlined
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<table>
<thead>
<tr>
<th></th>
<th>Pre-repair</th>
<th>Post-repair</th>
<th>% Recovered</th>
</tr>
</thead>
<tbody>
<tr>
<td>Maximum Load (N)</td>
<td>941.50</td>
<td>572.06</td>
<td>60.76</td>
</tr>
<tr>
<td>Stiffness-(m(N/mm))</td>
<td>163.64</td>
<td>149.69</td>
<td>91.48</td>
</tr>
</tbody>
</table>

Table 6.3: Results for 40% load drop criterion for specimen A4 scenarios, it is explicit that further investigation should be carried out using the percentage load drop off scheme.

![Graph](image)

Figure 6.15: Pre and Post-repair Load-deflection for specimen A4

**Stress Debilitation**

The specimens for this group were labelled \(F1, F2\) and \(F3\). A value of 75% peak load of the undamaged specimen \(A4\) was chosen as the ultimate load for each cycle. For specimens \(F1\) and \(F3\) it was 750 N, and specimen \(F2\) had a peak load of 700 N up to the 30th cycle, but was reverted to 750 N for the remainder of the
6. SELF REPAIR TECHNIQUE - THE HEALING RESPONSE

test. The stiffness, in terms of the slope of the load-deflection curve (henceforth referred to as ‘stiffness-m’) was computed for each cycle using the secant method, between the origin (0,0) and the load-deflection data at 700 N, until there was a 10% reduction in the stiffness-m. Thereafter 0.3 ml of Loctite 406 was injected through the unsealed hollow inlet. The CA adhesive was observed flowing out from the sealed end of the opposing inlet channel, which has a small void section near the blocked hole. However, no seepage of the adhesive was detected from the transversely oriented hollow section to the longitudinal inlet channel.

![Graph showing stiffness-m vs. loading-unloading cycle]

Figure 6.16: Specimen F1: Change in stiffness form. The post-repair stiffness-m shown in red asterisk

Specimen F1 was taken through the fatigue run for 23 cycles before it was infused with the healing agent. As Figure 6.16 shows, the laminate’s stiffness gradually decreases as the loading-unloading cycle increases before the test terminated after the 23rd cycle. At this point, 10% of the material’s stiffness had been lost. This means that as the loading cycle increases the level of internally induced damages
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also rises. This is evident from the profile of the material’s behaviour in Figure 6.16. The result of the accumulated damages was observed on the last test run. On this run, the test was stopped at 700 N as the load began to drop off. Subsequent examination revealed flexural damage at the mid-span. The post-repair outcome showed good recovery in that the stiffness-m of the post-repair test is approximately 26% less than its corresponding value at the undamaged state. This represents 74% recovery in materials efficiency. Further fatigue runs on the repaired specimen F1 were not carried out. The specimen failed in flexure, at mid-span, at 600 N load, on its first post-repair run.

Figure 6.17: Specimen F2: Gradual changes in stiffness-m from loading and unloading cycles. The post-repair stiffness-m for three consecutive runs are those shown in legend of: red square, green diamond and purple triangle.

The result presented in Figure 6.17 for specimen F2 shows the stiffness-m of all run cycles including those for the post-repair stiffness-m. The plot shows quick reduction in stiffness-m within the first eight runs. This was noticed at the early stages as superficial damages began to develop around the loading edge area, Figure 6.18. This can be attributed to the effect of the knife-edge on the
compression surface of the specimen all through the test runs. Repeatedly, the knife-edge crushes and ruffles the matrix along the loading line without inflicting any noticeable flexural damage on the specimen. The top surface damage exposes the immediate glass fibres underneath the matrix. This is considered a serious damage in real world applications as it has the propensity to weaken the structure from exposure environmental elements, cause further fibre-matrix debonding, delamination etc. This highlights the only major downside observed for the adopted fatigue method of testing using three point bending.

Figure 6.18: F2: Superficial damage—crushed matrix and fibre exposure—from the loading knife-edge

With small variation, F2 appears to have an average stiffness-\( m \) of approximately 131 N/mm between the 10th and the 30th cycle before the sudden decline as shown in Figure 6.17. No significant recovery was shown by any of the post repair loading-unloading runs. The stiffness-\( m \) at test conclusion remained approximately 10% below its pristine value. In other words, there was no explicit self healing in the material. Nevertheless, progressing declination of the stiffness-\( m \) was aborted as a result of the adhesive filling the nearby microcracks. Also, the three post-repair runs showed similar residual strength indications. These suggest that the specimen had experienced minor damage(s) either in the reinforcement, the glass fibres, or in the polymer matrix. It was devoid of sufficient energy for further damage or crack propagation necessary for proper connection with the hollow channels. For non-detrimental damage(s), higher proportion of the stress is shared by other healthy areas. This load sharing responsibility is explored more
later. Since there was an outward seepage of the adhesive at the opposite end of the inlet, it can be said that no damage occurred within the hollow channels’ proximity. This did not facilitate the healing of the affected areas, as it would probably have been in the case of substantial flexural fracture of the bottom glass fibres.

Specimen F3 appeared to be slightly stronger than F2 (by approximately 6 N/mm) at the first run for both specimen. F3 appeared to have good stability within its elastic limit for the first sixteen cycles with an average stiffness-m of approximately 142 N/mm, Figure 6.19. The succeeding four runs, specimen F3 marginally lost its elasticity by approximately 4.5% from the pristine specimen. This reduced stiffness can be attributed to the small flexural damage of the bottom layer, at the point of maximum flexure during the pre-repair penultimate cycle. The test was aborted on the 21st cycle at 620 N as the load began to drop off. During the repair, only 0.2 ml of 0.5 ml of the adhesive was successfully injected into the specimen, and no outward seepage was observed as it was in the case of specimen F2.

The plots in Figure 6.20 represents the load-deflection comparison between the pristine, final pre-repair and the post-repair state of specimen F3. It appears that there was less flexural stress at the mid-plane at the hollow network location. Therefore, less damages were experienced at this site. This indicates that the accumulating damages from each consecutive loading cycle caused a repair system isolation from the damage sites. This was possibly due to less significant impact of the accrued stresses in breaching the dividing walls of glass fibre-polyester matrix layers. This further increases the pressure at the injection point as the adhesive was gently pressurised into the specimen. Ideally, connections between the hollow network and the damage sites should create low pressure zones within the specimen. This would ease the diffusion of the injected adhesive from the higher pressure reservoir (the syringe in this case) to a low pressure damage sites. This explains why a fraction of the adhesive was injected. As a result, F3 showed no improvement in its elasticity with approximately 122 N/mm in stiffness-m for
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Figure 6.19: Specimen F3: Changes in stiffness-m from loading and unloading cycles. The post-repair stiffness-m is indicated with the red coloured “x” legend.

post-repair run dropping down from 128 N/mm for preceding run, F3-21, Figure 6.20.

Summary
The stress debilitation specimens showed no improved mechanical properties upon the influx of the CA adhesive into the hollow channels. In some cases, further reductions were observed. This degradation in the mechanical response is attributed to difference in pressure inside the specimens at the injection time and the pressure at which the adhesive was released from the syringe. Also, the degradation or lack of recovery in stiffness can be attributed to the ineffective functioning of the adhesive within a load range of 500-700 N. As it will be shown in the diminishing loading capacity test results, the CA adhesive is more effective below the stated load range. The peak load involved in the stress debilitation impacted sufficient damaging force on the cured CA bonds, enough to yield the bond formed between the CA elements and the glass-polyester substrates. This is not substantiated in this thesis as it requires appropriate non-destructive evaluations to indicate the
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spread of the CA adhesive in the specimens, at the damage sites, and at adjacent layers to the hollow network's position. Such evaluations were not carried out.

**Diminishing Loading Capacity**

*Pre-repair*

The group of specimens tested for this scenario were labelled LD1, LD2, LD3, LD4, and LD5. A pre-set test shut-down of 25% drop from the maximum load was used for LD1 and LD2, 30% for LD3, and 45% for both LD4 and LD5. The changes in the values were due to the erratic response of the Zwick's load cell. The three-point bending test was also repeated after the specimens were deemed to have failed. This was to characterised the specimens' mechanical capabilities at their failed states. The test results are shown in Figures 6.21, 6.23, 6.24, 6.25, & 6.26, and in Table 6.4. It can be observed from displayed figures and the table that the specimens were of similar mechanical strength at their primal condition. An average maximum load of 960 N and stiffness-m of 150 N/mm with standard deviations of 90.39 N and 12.34 N/mm respectively were recorded. The low stan-
standard deviation of the specimens’ stiffness shows they replicate similar responses within their elastic limits. From the analysis of the stiffness through the slope term, i.e. stiffness-$m$, $LD_4$ appeared to be the stiffest specimen at 167 N/mm and $LD_2$ the least at 138 N/mm. Before the commencement of the healing procedure, each specimen was examined. They had similar indentation on the compressive side caused by the concentrated loading by the knife edge. It was observed that all the specimens failed under two different modes—delamination for $LD_1$ and flexural failure of different pedigree for other specimens.

![Figure 6.21: LD1: Load-deflection curves at different repair stages](image)

All specimens except $LD_1$ had a flexural failure in the bottom layer, with a small developed curvature, and protruding fractured glass fibre strands at the mid-span. The severity of this damage on specimen $LD_3$ is illustrated by the “$LD_3$ at failure” plot in Figure 6.23. The residual strength in the specimen at failure and before post-repair test is considerably lower than those for $LD_1$ and $LD_2$ at their failed states. Delamination in $LD_1$ was situated in the mid-plane, same plane as

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### Table 6.4: Results for the load diminishing criterion

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Pre-repair</th>
<th>Post-repair</th>
<th>% Recovered</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Max Load (N)</td>
<td>Stiffness-m (N/mm)</td>
<td>Max Load (N)</td>
</tr>
<tr>
<td>LD1</td>
<td>902.47</td>
<td>157.39</td>
<td>848.64</td>
</tr>
<tr>
<td>LD2</td>
<td>848.64</td>
<td>157.39</td>
<td>848.64</td>
</tr>
<tr>
<td>LD3</td>
<td>1081.29</td>
<td>139.04</td>
<td>310.24</td>
</tr>
<tr>
<td>LD4</td>
<td>1007.51</td>
<td>166.73</td>
<td>300.21</td>
</tr>
<tr>
<td>LD5</td>
<td>957.92</td>
<td>146.52</td>
<td>473.71</td>
</tr>
<tr>
<td>A4</td>
<td>941.50</td>
<td>163.64</td>
<td>572.06</td>
</tr>
<tr>
<td>SD(LD1–5)</td>
<td>90.39</td>
<td>12.34</td>
<td>226.36</td>
</tr>
<tr>
<td>SD(all)</td>
<td>81.19</td>
<td>12.44</td>
<td>204.41</td>
</tr>
</tbody>
</table>

SD is the standard deviation.

It was observed that the entire volume (0.5 ml) of the adhesive quickly percolated through, and was observed flowing into the porous delaminated areas. The injection point lying on the left side of the support was unaffected by the delamination and was used as the supply channel for the healing agent. The delaminated areas created low pressure zones, an attraction for the pressurised injected fluid, unlike the hollow network, which led to adjacent layers’ separation. This type of failure can be attributed to weak matrix bonding between the layers and air voids presence during fabrication. The delamination covered half of the span-length of the specimen, from the centre propagating to the right hand support as shown in Figure 6.22. The load-deflection plot for LD1, Figure 6.21, at pre-repair stage does not suggest any gradual growth or propagation of delamination in the specimen. Otherwise, the two separated halves would have resulted into reduced stiffness due to infringement (voids) on the stress transfer pathway(s). This interlamina failure prevented more damaging failure involving the reinforcing fibres and the matrix fracture—flexural failure. This suggests that, in the presence of delamination initiator, failure would occur at slightly lower load (considering the loading capacity of the other specimens) than the material’s ultimate capacity.
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Figure 6.22: Delamination failure of specimen LD1. The red line highlights the delamination path

the specimens from the previous group, Section 6.7.3.

Post-repair

The general behaviour of the laminates after repair shows some resemblance of the pre-repair attributes especially in terms of stiffness. The stiffness of the post-repair specimens, within the linear portion (0-500 N), or otherwise stated range, showed an impressive recovery 128 N/mm on average. This represents 85% recovery with a standard deviation of 22.31 N/mm. On the contrary, the average mechanical loading capacity for the laminates upon repair showed a dramatic reduction of 47%. This comes with a standard deviation of 226.36 N for average maximum loading of 503 N. This high variation can be ascribed to different factors which are discussed in the following paragraphs.

Post-repair LD1 showed significant recovery in mechanical properties—loading capacity and stiffness. Recovered efficiencies were 94% and 98% for maximum load and stiffness-m respectively, Table 6.4. Examination of the failed post-repair LD1 showed no sign of delamination but flexural damage at half span length of the specimen. Therefore, delamination was completely averted due to the presence of tougher binder between the initially delaminated layers. Also, due to the initial (pre-repair) mode of failure—delamination, majority of the reinforcing fibres were unaffected, they experienced no breakage or fracture until the post-repair test. This explains how LD1 performs well above the other specimens due
to the difference in failure mechanisms. The near impeccable mechanical response of this specimen appears to the significant outlier, creating an overshoot for the standard deviation. With the exclusion of the LD1 from the statistical variation, the standard deviation reduces from 226.36 N/mm to 136.29 N/mm.

The performance of LD3, Figure 6.23, and LD5, Figure 6.24, bears similar traits to the model specimen, A4, in their failure fashion. The adhesive showed a commanding restorative capability in re-establishing the stiffness within the limit of half of the specimens’ initial loading capacities. The stiffness-m for both pre and post-repair was based on the load-deflection of the two specimens between 0-500 N. The stiffness-m of the mechanical properties of LD3 was recovered back to pristine state, while LD5 recovered approximately 91% of its initial stiffness, Table 6.4. On the contrary, the recovered loading capacity of both specimens was approximately 50% of their respective initial ultimate loading capacity. This 50% loss in the mechanical loading strength can be attributed to the fractured glass fibres in the pre-repair test. However, the gained loading strength denotes an interesting recovery as the load at 4 mm deflection, 540 N for LD3, is seven times the load magnitude at the same corresponding deflection at failure. This analysis shows that the adhesive influences the structural responses of the specimens. The maximum recorded failure loads upon repair show that the strength of the repaired specimen is singularly dictated by the adhesive’s bond strength when significant damages have been done to the reinforcing fibres.

During the repairing-healing process, injected adhesive was observed flowing out from the flexural damaged sites for LD3. This was not the case for LD5. At its (LD3) failure state, a few glass fibre strands, protruding out from the damaged site, were noticed. During the adhesive injection process, an outward flow of the repairing agent was observed from the one of the blocked transverse hollow inlet section, closer to the injection point. There was no observable evidence of an adhesive-drenching, or an adhesive-infusion at the damaged site. Hence, the truncated ultimate loading capacity can also be associated with the inadequate volume of the adhesive at the damage location. On this occasion, on a lesser
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Figure 6.23: LD3: Load-deflection curves at different repair stages

Figure 6.24: LD5: Load-deflection curves at different repair stages
degree in comparison of the two post-repair plots, the inadequacy of the healing-agent’s volume can be linked to the ‘inner pressure effect’ due to the absence of distinct linkage between the fracture paths and the hollow channels.

The inner pressure effect adversely affected the post-repair mechanical behaviours of specimens LD2 and LD4 as their respective load-deflection plots indicate in Figure 6.25 and Figure 6.26. The positioning of the post-repair plot in each figure explicitly indicates the occurrence of partial healing in the materials. As the figure shows, on the proportion of the linear phase, LD4 appears to be the less affected of the two specimens. The post-repair plots clearly indicate less significant recovery for both in either of loading capacity or stiffness. The maximum loading capacities were reduced to approximately 300 N, representing over 60% reduction and 70% for LD2 and LD4 respectively. The same is true of the stiffness-m which was estimated over a range of load values different from those used for LD1, LD3, and LD5. Due to the apparent disparities between the profiles of both pre and post-repair for LD2 and LD4, their ‘stiffness-m’s were evaluated between 0 N and 250 N, half the range used for other specimens, 0-500 N. This range was adjudged to be most linear portion for each post-repair plot. Half the range used the for pre-repair cases (0-500 N) was chosen for the post-repair plot (0-250 N). Over this short range, the specimens appeared to have lost approximately 20% (LD2) and 21% (LD4) in stiffness. This can be attribute to the high pressure - low pressure effect in diffusing the healing agent into the damaged parts and paths as reported in Section 6.7.3. Only a small fraction of the adhesive was successfully injected into the specimen, hence the small recovery shown in Figure 6.25 and in Figure 6.26. The pressure created inside the material during repair arises due to improper or inadequate connection between the damage path(s) and the nearest hollow channel.

The small recovery in loading capacity and stiffness was somewhat expected. The fracture from the flexural damage was lacking adequate intensity to create a damage path for proper connection between the hollow network layers and the lower adjacent layers. The flexural damage at the outermost layer showed that inner
6. SELF REPAIR TECHNIQUE - THE HEALING RESPONSE

Figure 6.25: LD2: Load-deflection curves at different repair stages

Figure 6.26: LD4: Load-deflection curves at different repair stages
layers would have less curvature from the induced bending moment. As a result, in the case of LD4, the adhesive was observed flowing out from the neighbouring unblocked hollow channel. This indicates that the entire volume of the network had been filled or debris (from the flexural fracture) had created blockages on the fluid’s path.

The recovery responses of these specimens (LD2 and LD4) were further investigated. The cross-sections of the specimens, at the flexural failed sites, were examined using a scanning electron microscopy (SEM). LD3 was also included as a reference specimen. Figure 6.27 shows the images of the specimens’ hollow section at their respective flexural damage locations (mid-span). Figure 6.27c shows a blocked hollow passage, which indicates the healing agent’s flowed into the major damaged area. Conversely, Figure 6.27a shows a clear image of an opened and unfilled hollow section, while it was blocked with the CA resin, 5 mm from the cut section. This denotes the healing agent had polymerised before reaching the failure point. This premature polymerisation is attributed to trapped air volume within the network, unable to escape through failure area. This created an imposing wall with sufficient humidity to activate the curing of the healing agent within few micrometer of contact. As it was for other specimens, large scale of structural damage yields better recovery than small scale damages. The efficiencies of the recoveries are a reflection of the vascules location in the specimens. In this case, the position was not at an optimised location to influence greater recovery.

Summary
It is quite clear that Loctite 406 adhesive used in redressing flexural damages has limited load range for such an application. This is especially true when it involves restricted human control. In such scenario, the adhesive is delegated with diagnostic duties of assessing damages and of self navigation to weakened spots, followed by cautious settlement on the area to bring forth healing. This task is quite demanding and would require a smart adhesive. Also in all the test, ductility was not improved upon repair. All failures were sudden with little warnings. However, despite the allowable load restraint on the adhesive, the
6. SELF REPAIR TECHNIQUE - THE HEALING RESPONSE

Figure 6.27: Micrographs of LD2 and LD3's cross-sections, showing the hollow sections at their flexural failure locations

The technique employed here showed that the hollow channels of single layer, in the mid-plane, at a safer distance from the vulnerable bottom layer, can be used to restore an average of approximately 46% of the loading capacity and 84% of the valuable stiffness parameter. These numerical figures exclude results from LD1 due to damage type differences but are inclusive of the results for specimen A4. This implies that both the restored mechanical strength in loading and stiffness can be improved through critical placement and use of multiple hollow channel layers. This has been demonstrated through the successful usage of the vascular network for self healing proposed, designed and tested in this chapter. Also, most importantly, the post-repair analysis shows that limitation of the damages done to the fibres is a significant factor that affects the efficient of the recovery for both load and stiffness. Fractured fibres operate differently and are less efficient.
Chapter 7

Progressive Ply Failure Analysis

7.1 Introduction

In the preceding chapters, it was shown that crack formation, the main delamination instigator, could arise as a result of generated localised stresses at certain section along the length of a WTB (Chapter 4). Also, in Chapter 6 weakened sections on self healing specimens, LDs and Fs were isolated from the healing agent supplying routes in the hollow network and consequently sufficient strength was not regained upon repair. This results from the isolation of susceptible section(s) from the hollow passage connections. In such a case, a failure predictive and diagnostic tool, highlighting the vulnerable layer(s) of an FRP composite structure can act as a guiding tool for permanent location of a self healing system. This chapter explains the formulation of such a failure predictive mechanism, purposed for target-positioning or strategic-placement of a self healing system in a FRP composite structure. Relevant literature on progressive ply failure analysis is present as well as the introduction of the PPFA model adaptation used in the subsequent section. The chapter closes with a comparison between the developed PPFA model, experimental results (Pre-repair, Section 6.5) and the conventional approach.
7. PROGRESSIVE PLY FAILURE ANALYSIS

7.2 Methodology

PPFA refers to the computational and the theoretical models currently used in the analysis of failure of fibre reinforced composites. The analysis aims to theoretically predict the failure order, location, and strength extinction states in a fibre laminated structure. The developed model will mirror the failure mechanism experienced under operating conditions. Increasingly, those at the highest demand chain for FRP composite materials would prefer robust and close precision analysis tools as a means of curtailing maintenance cost, taking preventive action before damages progress to an unserviceable state, and thereby avoiding sudden failures. Such a tool would produce, structural responses such as stress and strain, relatively close to that of real structures from internally or externally induced stimuli, starting from damage initiation stage to ultimate failure. Computational finite element (FE), has formed the bases for the rapidly advancing and cost effective method of structural failure predictions through an avenue of damage monitoring in FRP composite materials.

Many methodological and theoretical procedures for PPFA have been well documented over the past two decades (Daniel, 2007; Fan et al., 2011; Kim et al., 1996; Tay et al., 2008) based on damage and fracture mechanics e.g. degradation variation and cohesive element respectively. Some of the widely used theories are the Maximum Stress theory, Maximum Strain theory, the Maximum Work theory (Tsai-Hill, and Tsai-Wu). They are popular due to their ease of use. Kim et al. (1996) performed an analysis to assess the redistribution of stress on the elements of a laminated composite beam. The study uses layer-wise constant shear in predicting failure stresses of elements using material degrading factors under two independent failure criteria—Maximum Stress and Tsai-Wu theory. Daniel (2007); Tay et al. (2008) have both reviewed studies published under progressive failures of FRP composites at different scales of analysis—macro and microscopic. Fan et al. (2011) examined the propensity of damage growth in an FRP sandwich composite under different simulated failure modes such as Hashin’s criterion. The damage evolution behaviour on the composite was model using the progressive failure analysis.
Most of the postulated theories currently utilised are based on the properties degradation method (PDM), as subversion of degradation variation which is commonly centred around the first ply failure (FPF) analysis. The FPF principle operates in a manner where a complex FRP structure exhibits local stress concentration zones, a failure originating area. Within each stressed zone is the internal architecture of a series of fibre in distinct stacking order and orientations. While there may be a common level of deformation for all the layers in the zone in question, the stress level in each, will however be different. The individual layer of reinforcing fibres will fail in order of their strength when the portioned, applied load, exceeds the layer’s ultimate strength capacity. From the classical lamination theory, the stiffness constitutive parameters at the first layer failure can be obtained for such laminate. It is experimentally challenging to replicate the results obtained from a numerical or FE model for FPF technique and vice versa. The major influencing factor on the strength of a laminate’s layer is the angular orientation of the fibres relative to the loading direction. Layers oriented parallel to loading directions are considerably stronger than those transversely oriented.

Among the aforementioned failure theories Tsai-Hill’s maximum-work theory is the most extensively used as the ultimate loading capacity can be directly obtained at design stage. Also, unlike the others, its results are based on interaction between the material’s mechanical properties. The other earlier theories have a drawback of poor prediction due to their non-interactiveness. The predictive quality is not only dependent on the chosen analytical method but it is also strongly influenced by the criteria defined for some underlying parameters such as what the ultimate laminate failure means, the quantification of resin polymerisation induced stress and impact of high matrix proportion on low fibre content laminates (Daniel, 2007).

The progressive ply failure analysis has evolved in the past decade from the earlier theories which many had used with the logic of permanently suspending the
stiffness properties of any failed ply or layer by changing them to zeros at the slightest failure detection (Agarwal et al., 2006; Pal & Bhattacharyya, 2007). In the test conducted by Greif & Chapon (1993) the application of this method was found to have significantly underestimated the laminate elastic modulus. The numerical prediction did not match the experimental results. However, it was found that convergence issues could be sidestepped by using smaller values of stiffness in the computational process (Apalak et al., 2006, 2007), a formal approach of progressive degradation method. Reddy et al. (1995); Reddy & Reddy (1993) have used the stiffness reduction coefficient (SRC) method with coefficient between 0 and 1. After the first indicated element failure and before each subsequent layer’s failure, a gradual reduction, using SRC, was applied to the elastic modulus until the failure criterion became insatiable.

In the investigation in this thesis, PPFA is used with a slight different logical proposition to that employed by Reddy et al. (1995). Their report uses gradual stiffness reduction on any damage element until the failure criterion cannot be satisfied. The modelling described in the subsequent section entails a macroscopic PFA at ply-level and not microscopic. An elastic modulus’ degrading factor, in the form of SRC or material properties degrading method (MPDM) (Prusty, 2005; Tay et al., 2008), is applied to the stiffness and once the damage presence has been certified in any failed layer by the chosen failure criterion, the layer in question is relatively hibernated in its role. The model follows some logical procedure in other to account for the residual strength upon failure. Introduction to classical laminate theory and general behaviour of reinforced fibre in composites under loading relevant to this discussion have been well published over the years, (Agarwal et al., 2006; Jones, 1999; Kaw, 2010).

7.3 Model Formulation

Since individual layer contributes a certain amount of strength to the global (laminate’s) stiffness, the failed lamina’s stiffness is gradually reduced through MPDM (Tay et al., 2008). Under real operational conditions, FRP composites fail from
compound failures of the constituting components in a progressive manner, however short it may be. The failure rates depend on the intensity and magnitude of the damaging source. MPDM assumes that at the first failure of an individual layer, there is a residual strength to contribute to the general stiffness of the laminate. MPDM reduces the layer’s longitudinal strength, transverse strength and the shear modulus by certain specified constant(s). This is different from other conventional approaches to PPFA where the residual stiffness of a failed layer is considered inactive. Such layer is adjudged to be structurally ineffective, and the stiffness matrix is zeroed or obsoleted. The MPDM described below uses a cyclic failure criterion where a layer, e.g. the $i$-th layer is deemed to be partially ineffective after $n$-number of failures. Prior to reaching the $n$-th failure detection for the $i$-th layer, the layer’s strength is reduced by the MPDM constant. At the $n$-th failure detection, the layer is demoted to a passive role. In such state, it is considered ineligible for further failure detection until other layers have failed $n$ number of times. Since the residual strength at $n$-th failure is retained, the $i$-th layer will also contribute to the global stiffness. Once every layer has failed $n$-time, the program is simply repeated for one more level of failure.

The numerical failure prediction simulation is carried out using Matlab, a mathematical tool. The program runs on the primary inputs: elastic modulus and constants; ultimate strengths; layers thickness and orientation angles; MPDM—reduction factors; failure criteria; and $n$, the number of failure per layer. The global stiffness matrix for the laminate is generated from the elastic modulus and constants $-E_L, E_T, \nu_{LT}, G_{LT}$. These are the elastic modulus in the longitudinal direction, in the transverse direction, the Poisson’s ratio and the shear modulus, respectively. The major stiffness matrix in the primary loading direction is in turn used to compute the local stiffness in each individual layer which are the essential inputs along with the corresponding thickness to obtaining the constitutive matrices $A - B - D$ in equation 7.1 of the laminate from a subsidiary algorithm.

$$
\begin{bmatrix}
F \\
\vdots \\
M
\end{bmatrix} = \begin{bmatrix}
A & B \\
\vdots & \vdots \\
B & D
\end{bmatrix} \begin{bmatrix}
e^0 \\
\vdots \\
k^0
\end{bmatrix}
$$

(7.1)
Where $A$ is extensional stiffness matrix; $B$, the coupling stiffness matrix; $D$, the bending stiffness matrix; $F$ and $M$ are the load and moment matrices; $\epsilon^0$ and $\kappa^0$ are strain and curvature respectively at the mid-plane. The extensional matrix controls the in-plane stiffness of the laminate, while the bending stiffness matrix, $D$, relates the resultant bending moment to the curvatures.

In the simulated three-point bending test, the laminate is set-up as it would be in a practical test. The laminate of dimensions width, $b$, and span length, $L$, is simply supported at both ends of the span. The laminate is loaded through displacement control at the midspan. According to the stress-strain relation, the maximum deflection, $\delta_{\text{max}}$ and the corresponding moment $M_{\text{max}}$ of a simply supported beam under a point load, $P$, at the midspan are given as:

$$\delta_{\text{max}} = \frac{PL^3}{48EI}$$

(7.2)

$$M_{\text{max}} = \frac{PL}{4}$$

where $EI$ is the stiffness which can be replaced by $bD_{11}$, the product of the width and first term from the bending stiffness matrix. Rearranging the deflection equation and substituting it into moment equation in equation 7.2 gives:

$$M_{\text{max}} = \delta_{\text{max}} \frac{12bD_{11}}{L^2}$$

(7.3)

The resulting moment is equation 7.3 is then passed on to another auxiliary numerical program that computes the resultant stresses in each layer in response to the applied moment. The output stress is then forwarded to the chosen Boolean model of the failure criterion, which validates the structural health of each laminate. Assuming failure was detected and provided the allowable number of failures $n$, has not been exceeded, the entire laminate stiffness model is updated based on the Boolean result, starting from the elementary level of updating the local
stiffness according to the MPDM factors to the global stiffness. At this point, the A-B-D matrices are again recomputed to obtain the new bending stiffness. In the event of no detected failure, both local and global stiffness remain intact and the displacement unit is increased. This process continues until each layer has been reported to have failed \( n \)-time.

This formulation can be used for lateral or normal loading. The bending moment in the matrix is equated to zero for the former, and vice versa for the latter. In the numerically simulated three-point bending test, the FRP material has layers with alternating orientation angles \([0^\circ/90^\circ]\), and the loading is applied uni-axially, normal to the material plane. That is, only the bending moment, \( M_x \) along the longitudinal direction is active while the rest \( M_y \) and \( M_{xy} \) are set to zero. The flowchart of how in the program is executed is shown in Figure 7.1.

### 7.4 Numerical Results

The model FRP composite considered here has the same dimensions as the specimens with drilled hollow channels in Chapter 6. The numerical FRP laminate is simply supported on a 100 mm span, 46 mm wide and has 1 mm thickness per layer. The model, a glass/polyester laminate, consists of 22 layers with \([0^\circ/90^\circ]\) stacking order. The listed properties in Table 7.1 and MPDM in equation 7.4 were arbitrarily chosen but are representative of the FRP composite's properties to certain extent. The MPDM matrix is applied at each ply failure to modify the stiffness matrix \((\overline{Q}_{ij})\) in equation 7.1. Also two levels of permitted failure per layer, \((n=2)\), was used.

\[
MPDM = \begin{bmatrix}
0.65 & 0 & 0 \\
0 & 0.65 & 0 \\
0 & 0 & 0.65
\end{bmatrix}
\]  
(7.4)

In the stacking order, ply No 1 and ply No 22 are the top and bottom layers respectively. Table 7.2 and Figure 7.2 illustrate the results obtained for a cross-ply laminate at two levels of failure. The figure compares the numerical load deflection
Figure 7.1: Flowchart for the PPFA-MPDM model for a simply support FRP beam
7.4 Numerical Results

<table>
<thead>
<tr>
<th>Properties</th>
<th>Strength value (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$E_L$</td>
<td>19.3</td>
</tr>
<tr>
<td>$E_T$</td>
<td>4.13</td>
</tr>
<tr>
<td>$G_{LT}$</td>
<td>4.14</td>
</tr>
<tr>
<td>$v_{LT} = 0.3$</td>
<td></td>
</tr>
<tr>
<td>$\sigma_{LU}$</td>
<td>1.274</td>
</tr>
<tr>
<td>$\sigma_{TU}$</td>
<td>0.078</td>
</tr>
<tr>
<td>$\tau_{LTU}$</td>
<td>0.072</td>
</tr>
</tbody>
</table>

Table 7.1: Material properties of the modelled cross-ply laminate

result with the experimental result of sample B from Section 6.5 in Chapter 6. Likewise, Figure 7.4 compares the model result with all the experimental samples from the Section 6.5. The discussion of the results is limited to the comparison between the experimental sample B (pre-repair) and the model result. Figure 7.2 shows the numerical model lacks adequate toughness and ductility compared with the experimental result, though they share almost similar deflection at lower loads. The plotted results show similar elastic behaviour between the numerical model and the experimental sample. This is evidence in the flexural stiffness values within the elastic range (4-15 kN) for both plots which yields approximately 205 Nm$^2$ and 197 Nm$^2$ in stiffness for the numerical model and the experimental test respectively. The major difference between the two lies in the ductility of the materials. The experimental sample exhibited early plasticity when compared with the numerical model changing into plastic mode around 15 kN. Conversely, the numerical model at same load remained elastic and failed under the same mode.

The primary point of interest remains the sequential order at which all of the layers fail. As expected the layers failed relative to their distances from the mid-plane for the model as illustrated in Table 7.2. Those above the mid-plane failed in compression while those below it had a tensile failure. For this laminate, the outermost layers are those exposed to highest tensile and compressive stresses hence their failure at the lowest deflection. This makes layer No 22 the FPF at a few load just above 1000 N. Table 7.2 shows the weaker layers, their positions and
7. PROGRESSIVE PLY FAILURE ANALYSIS

<table>
<thead>
<tr>
<th>No of failure</th>
<th>Layer No</th>
<th>Angle (degrees)</th>
<th>Failure Load (N)</th>
<th>Deflection (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>22</td>
<td>90</td>
<td>1051.8</td>
<td>0.2471</td>
</tr>
<tr>
<td>2</td>
<td>2</td>
<td>90</td>
<td>1210</td>
<td>0.2471</td>
</tr>
<tr>
<td>3</td>
<td>20</td>
<td>90</td>
<td>1221.2</td>
<td>0.3091</td>
</tr>
<tr>
<td>4</td>
<td>4</td>
<td>90</td>
<td>1503.7</td>
<td>0.3091</td>
</tr>
<tr>
<td>5</td>
<td>18</td>
<td>90</td>
<td>1516.7</td>
<td>0.4121</td>
</tr>
<tr>
<td>6</td>
<td>6</td>
<td>90</td>
<td>2020.2</td>
<td>0.4121</td>
</tr>
<tr>
<td>7</td>
<td>16</td>
<td>90</td>
<td>2073.7</td>
<td>0.6171</td>
</tr>
<tr>
<td>8</td>
<td>1</td>
<td>0</td>
<td>2363.3</td>
<td>0.6171</td>
</tr>
<tr>
<td>9</td>
<td>8</td>
<td>90</td>
<td>2363.3</td>
<td>1.2341</td>
</tr>
<tr>
<td>10</td>
<td>21</td>
<td>0</td>
<td>2218.8</td>
<td>1.2341</td>
</tr>
<tr>
<td>11</td>
<td>14</td>
<td>90</td>
<td>2086.5</td>
<td>0.1611</td>
</tr>
<tr>
<td>12</td>
<td>3</td>
<td>0</td>
<td>2304.3</td>
<td>0.1781</td>
</tr>
<tr>
<td>13</td>
<td>19</td>
<td>0</td>
<td>2262.5</td>
<td>0.1851</td>
</tr>
<tr>
<td>14</td>
<td>5</td>
<td>0</td>
<td>2554.7</td>
<td>0.2251</td>
</tr>
<tr>
<td>15</td>
<td>17</td>
<td>0</td>
<td>2600.9</td>
<td>0.2421</td>
</tr>
<tr>
<td>16</td>
<td>10</td>
<td>90</td>
<td>3373.9</td>
<td>0.3231</td>
</tr>
<tr>
<td>17</td>
<td>7</td>
<td>0</td>
<td>3424.5</td>
<td>0.3281</td>
</tr>
<tr>
<td>18</td>
<td>15</td>
<td>0</td>
<td>3583.7</td>
<td>0.3501</td>
</tr>
<tr>
<td>19</td>
<td>12</td>
<td>90</td>
<td>4148.5</td>
<td>0.4141</td>
</tr>
<tr>
<td>20</td>
<td>9</td>
<td>0</td>
<td>5770.5</td>
<td>0.5781</td>
</tr>
<tr>
<td>21</td>
<td>13</td>
<td>0</td>
<td>6388.9</td>
<td>0.6441</td>
</tr>
<tr>
<td>22</td>
<td>11</td>
<td>0</td>
<td>23977.1</td>
<td>2.424</td>
</tr>
</tbody>
</table>

Table 7.2: Three-point bending test result for a 22 layers modelled cross-ply laminate

their failure order. The table also reports on consecutive order at which majority of the transversely oriented layers fail. The failure can be attributed to the large angles of the layers relative to the loading direction and their stacking positions. Those closer to the mid-plane enjoyed relative serenity. The furthest layers are more prone to early failure and same is true for the longitudinal layers. Based on the stacking sequence of the modelled laminate, the \([0^\circ]\) layers appeared stronger mainly due to their orientation with the loading direction.

There is no clear indication in Figure 7.2 to ascertain the point at which weakened layers failed in the experimental sample. Few slight drops in load around 3 mm...
7.4 Numerical Results

Figure 7.2: Comparison of numerical three-point bending result with experimental test result for a cross-ply laminate

deflection could be viewed as a failure signal in some layers. Likewise, it can be attributed to the effects of deep-seated defects such as voids. However, in the numerical model, majority of the layers failed in the early part of the test as Table 7.2 shows. This is further iterated by a point-plot at each failure point in Figure 7.3. The last points before the drop are the first and second failures of the 11th layer. The high failure strength of the 11th layer proves the significance of the residual strength of the previously damaged layers to the material strength.

7.4.1 Conventional Method

Progressive failure occurs from instability or shift in a structure’s equilibrium as a result of unexpected addition of extra loads. One common modelling technique for this occurrence is the conventional method. The conventional approach is considered as a pragmatic method in structural applications of modelling progressive failure of stacked polymer layers or progressive collapse of multi-storey buildings. This method deems a failed layer, either caused by the reinforcing fibres or by the
Since studies remain inconclusive on the rationale reduction on the elastic constant of any failed layer, the properties of such damaged layer is zeroed for the subsequent computations. Any failed layer is considered dormant at first failure detection and the respective stiffness matrix is subsequently zeroed. The unquantifiable residual strengths in the failed members are adjudged hypothetical and thus ignored. This can be regarded as a conservative method of predicting the allowable loading capacity. The inconclusiveness arises from the failure type, failure in the longitudinal fibres considerably weakens the transverse fibres, while failure in the former does not drastically affects the strength of the longitudinal fibres. However, under this traditional approach of modelling both classes of failure, either major or minor carries the same degree of severity.

The results of the conventional modelling of the cross-ply laminate in Table 7.1
7.4 Numerical Results

Figure 7.4: Comparison of numerical three-point bending result with other experimental test results

is shown in Figure 7.5. The plot exhibits many sharp peaks. Each peak, between 2000-2500 N, represents failure of a layer. Generally, the results completely underestimates the residual strength in each layer after failure.

The model shows that the conventional method of lamina withdrawal upon failure completely underestimates laminate strength. The MPDM approach on the other hand does not accurately account for the laminate’s post-failure residual strength but provides close comparison with the experimental result. The model also showed that under the induced bending moment, the vulnerability status of potential FPFs depends first, on the orientation of the laminate layers and second on their relative distance from the mid-plane. From a self healing viewpoint, the most interesting aspect is to locate the FPF layer and to have the certainty that it will not be shifted to other layers, regardless of its residual strength after the first failure. Through the numerical model, the critical layer(s) in a self healing system can be identified along with the corresponding load and deflection.
7. PROGRESSIVE PLY FAILURE ANALYSIS

Figure 7.5: Three-point bending result using conventional modelling approach

Figure 7.6: Indication of failure load and deflection
Chapter 8
Self Healing Structure

8.1 Introduction
This chapter encompass all findings from the previous chapters culminating into a single experimental entity. Stress concentration investigation in Chapter 4 revealed the susceptible region along the length of a WTB. In Chapter 5, the delamination failure in composite was experimentally examined using DCB tests. It was followed up with an investigation into self healing system for small composite structure. Furthermore, Chapter 7 explained through a numerical model how most vulnerable layer can be identified in such region. This penultimate chapter brings together topics discussed in preceding chapters. A flexural test conducted on a 500 W in-house built WTB is reported in this chapter. The blade contains an embedded 2D vascular network at a location, one third length of the blade’s length from the root. Only the suction half, the upper part of the WTB was made and analysed.

8.2 NACA 4415 WTB’s Features
Small size wind turbines are often commonly operated with blades of NACA 4415 aerofoil model. Some of these turbines have permanent magnetic generator (PMG) as the energy generating house in place of the nacelle, (Campos et al., 2001; Mishnaevsky-Jr. et al., 2011). At 5 m/s wind speed such turbines are
8. SELF HEALING STRUCTURE

capable of generating up to 80 watts at 286 rpm. The blade itself is 700 mm in length with chord length of 168 mm and 68 mm at the root and tip respectively. It was chosen due to the suitability of its short length for the Zwick Roell test rig (keeping a similar testing method—three-point bending test), and for ease of fabrication—linearity at both leading and trailing edges from the root to the tip. Also, it has less complex twist along the length of the aerodynamic shell. Aside the physical characteristics, NACA 4415 along with NACA 4412 blades have been reported to have some aerodynamic advantages over other aerofoil types. The profile enables more efficient operation of the turbine in terms of higher power output, a result of coherent relation between the power coefficient, $C_p$, to the tip-speed-ratio (TSR) (Maalawi, 2011). Furthermore, Kurtulumus et al. (2007) reported the lift rate at a set angle of attack for NACA 4415 blade to be much higher than those of NACA 0012 and NACA 23012.

8.2.1 Two-dimensional Hollow Network

The experimental results and the numerically modelled results from Chapter 6 and Chapter 7 respectively, explicitly showed that progressive failures arising from stress accumulation have to be counteracted at their on-setting layers—outermost layers, at the third length for a structure like WTBs, Section 4.2. The 2D hollow networks in the GFRP NACA 4415 blade were strategically placed at the 2nd and the 6th layers from both top and bottom of the blade. The network was centred at 230 mm length of the blade from the root and covers an area of 0.025 m$^2$. Each level of the multiple hollow network was formed by connections between six evenly spaced longitudinal hollow spaces and five transverse hollow spaces, Figure 8.1. A temporary formwork, in this case, a 450 $\mu$m diameter nylon cable (fishing line) was used to form each hollow space. The fishing line material was chosen due to its high loading capacity of 110 N. The cables were extracted after the laminate had cured. The network's coverage is the area enclosed by the intersections of the longitudinal and the transverse channels.
8.3 Fabrication of NACA 4415 WTB top-half

NB: The dimensions shown for the blade's ends (root and tip) are the arc lengths of the upper surface of the aerofoil at both ends.

Figure 8.1: NACA 4415 WTB 450 μm hollow network layout

8.3 Fabrication of NACA 4415 WTB top-half

A wooden replica, Figure 8.2 was made from a piece of timber. The timber was divided into 14 stations and each division was chiselled down to its appropriate NACA 4415 profile. Thereafter, the prototype was placed in a silicone based moulding agent (Mold max-30), on which the top-half of the blade was imprinted. The mould mixture was allowed to cure for 24 hours before the wooden blade was taken out. The edges of the silicone mould were subsequently redressed to enhance workability.

Figure 8.2: Wooden NACA 4415 WTB prototype

The WTB was fabricated in a similar fashion as the GFRP coupons in the earlier
8. SELF HEALING STRUCTURE

chapters. Fifteen layers of woven E-class, 200 g/m² glass-fibre, were cut into a trapezoid like shape of the blade. In general, most small WTBs are isotropic in their mechanical properties and for this feature to be present, eight of the layers were cut in their primary orientation of [0°/90°]. The remaining seven were cut out such that they were at [+45°/−45°] to their primary orientation. The blade was fabricated with alternating layers of the stated orientations. A 630 g of Norsodyne H 13212 TAE polyester resin (GRS) was apportioned in a 2.5:1 ratio by weight with the glass fibres according to the manufacturer’s recommendation. Also, 12.6 g of organic peroxide methyl ethyl ketone peroxide (MEKP) was added to the polyester resin as a polymerising agent.

The silicone mould and the nylon cables were coated extensively with a demoulding agent to facilitate post curing detachment of the blade from the mould and removal of the nylon wires from the GFRP blade. Afterwards, a small volume of freshly prepared polyester matrix was spread evenly on the mould using a paint brush followed by a careful placement of the first layer [0°/90°]. This procedure was repeated for the second layer but with the layer oriented as [+45°/−45°]. At this point the nylon cables which had been attached to balsa wood (at one end), at a specified distance from one another were placed on the matrix soaked glass fibre clothing. The wires were held in position via slits cut into the mould at the appropriate positions as shown in Figure 8.3. The layering process was resumed along with alternating fibre orientations and was only stopped for the cables’ placements at the 6th, 9th, and 13th layer. The blade was allowed to cure for 24 hours in the mould at room temperature after which it was removed.

The GFRP blade was further cured for the same period and under the same conditions. Afterwards the transversely placed nylon wires were removed. However, initial attempts at drawing-out the longitudinal wires failed due mass contraction of the matrix on the cables. The blade was heated in a Memmert oven at 80 °C for 45 minutes to reduce the contraction stress. This thermal treatment, which was below the degrading temperature (Avila et al., 2007), allowed the coated material
8.4 Mechanical Tests

The fabricated blade was first characterised through a simple cantilever beam test to obtain its stiffness. Afterwards, it was set up for a flexural bending test. In the characterisation, the root of the blade was rested in between two timber frames which were clamped together to a steel structure, Figure 8.4. The tip deflection was measured using a linear variable displacement transducer (LVDT) as the load, which was placed 600 mm from the inner edge of the support, was manually increased. Readings were recorded for every 5 N increase in load. The test was stopped at a load of 25 N.

The elastic stiffness, $EI$ was calculated using equation 8.1 which is obtained from the rearrangement of maximum deflection equation for a cantilever beam with a tip load.

$$EI = \frac{P L^3}{\delta 3}$$  \hspace{1cm} (8.1)
The expression, $\frac{P}{\delta}$, can be taken as the slope from the linear plot of the load against deflection values obtained from the test. The length, L (mm) is the measured distance between the loading point and the support.

The flexural bending test was carried out using the Zwick Roell testing machine. The set-up is as shown in Figure 8.5. The test was conducted in displacement control mode at a rate of 2 mm/min with a 10 kN load cell on the previously used Zwick Roell. A maximum load limit and extension of 2 kN and 30 mm respectively were used. The automatic shut down threshold was set to 50% drop in peak load. The test was conducted such that the load was held on the blade after test termination. The blade was simply supported at the root and at 57% of its length by two timber frames with a 300 mm span. The wooden blocks were sculptured to fit the blade's cross-sectional profile at their respective positions. This...
8.4 Mechanical Tests

arrangement enabled the loading head of the Zwick Roell to be directly above the centre of hollow network passages. The blade was loaded from the inside rather than from the outside (the suction side), Figure 8.5. Also in a similar fashion to the supports, a small timber piece was placed at the loading point for load distribution across the blade’s width. The blade was slightly trimmed at the root section by 5 mm to prevent curvature which had developed from fabrication from hindering the adhesive passage during repair. The cut surface was subsequently cleaned with an acetone. All the transverse hollow outlets were not used as the repairing agent’s inlet points and were blocked with a silicone gel.

Figure 8.5: Set up: Flexural testing of NACA 4415 WTB

The gel was allowed to set and cure for 2 hours before the flexural testing. This test was carried out for three distinct stages:

- the blade is tested to failure
8. SELF HEALING STRUCTURE

- test is repeated to confirm failure
- post-repair test is conducted

A positive failure confirmation test denotes repair commencements. In this test, the blade was injected with Loctite 406 using the topmost longitudinal hollow inlets at root as the injection points. The injected adhesive was allowed to cure for 24 hours at room temperature. Afterwards, the post-repair test was conducted. The efficiency of the repair scheme was analysed based on the effective flexural stiffness, \((EI)_{eff}\) of the blade at both stages of the testing. This is the same analytical approach used in Section 6.4 with equation 6.2.

Generally, WTBs are known to be of non-uniform stiffness due to their tapered profile. However, in the analysis of this test, the NACA 4415 blade is assumed to be of uniform cross-section and therefore uniform stiffness. This is not considered a standard test for WTBs as those discussed in Chapter 4 but was contrived to grade the blade’s flexural responses under a damaging load. Also, the influence of the repairing agent on the blade's stiffness can be quantified.

8.5 Results and Discussion

Pre-repair and Adhesive inflow

The undamaged effective stiffness characterisation plot obtained from the cantilever test is shown in Figure 8.6. Using the slope extracted from the plot by the least square method, approximately 72 Nm\(^2\) was calculated as the effective stiffness at its undamaged state.

The immediate post-test visual examination following the damaged confirmation test, revealed a delaminated area, Figure 8.7, near the loading head of the testing apparatus. This damage is located between the second and third layers below the loading head. This is attributed to the presence of the hollow network not matching the curved profile of the layers. As a result, more resin was used in the fabrication process, which created a weakened section between the layers. The
8.5 Results and Discussion

Pre-repair load-deflection plots are presented in Figure 8.8. The figure shows the blade’s behaviour from its elastic range to failure indentation indicated by the apparent non-linear deformation in between 8 mm and 25 mm. The other plot, ‘at failure’ confirms initiation of damage into the blade. It (‘at-failure’) shows a complete deviation from the initial plot, ‘pre-repair’, right from the loading onset. It appears that permanent non-linear deformation had been induced into the blade as shown by the distinctive elastic behaviour of the blade at the ‘pre-repair’ and the ‘at failure’ test. This is further iterated by the elastic flexural stiffness values of approximately 87 Nm² and 55 Nm² calculated at a load of 1000 N for both the ‘pre-repair’ and the ‘at failure’ curves respectively, using the secant modulus as shown in Figure 8.13. The difference in the flexural stiffness values shows that the blade had lost approximately 35% of its flexural stiffness.

![Figure 8.6: Load-deflection plot for elastic stiffness](image)
Furthermore, the deformation around the average peak load of 1800 N of the 'pre-repair' plot defines the damage state of the blade and as a result, the test was ended by the extension limit. This cannot be seen from Figure 8.8 due to the large amount of data accumulated exceeding the limit of the Microsoft Excel processor. The change in the flexural failure mode from sudden load drop to non-linear deformation in the plot can be attributed to the manner in which the load was being distributed. There was no flexural failure in the light of the bottom layers failing first as it was in Chapter 6, where the flexural failure was caused by the applied concentrated knife edge load. However, the timber block employed allowed the load to be further spread around it. Consequently, this led to the delamination around the weaker region of the blade near the loading timber and the trailing edge, Figure 8.7. The failure cause is not definitive but can be
attributed to a wider spread of stress at the loading point and the presence of the hollow network. Also, the asymmetric geometry of the blade can be said to have contributed to this damage due to the stiffness discontinuity.

A total volume of 5.5 ml of Loctite 406 was manually injected into the blade while the load was still held on it. The load was taken off after injection had been completed. The influx of the adhesive was started from the first injection point, in the second column near the trailing edge. This was to enable the adhesive a short travel route to the delaminated site. Droplets of the adhesive were observed at the nearest previously sealed transverse hollow section in the adhesive’s path. This confirms that both the transversely and longitudinally oriented hollow sections are well connected and that the transverse hollow passages were
8. SELF HEALING STRUCTURE

not sufficiently sealed by the silicone sealant. Also, outward seepage of the adhesive was observed at the neighbouring injection points. The adhesive quickly covered a large area of the injecting surface making it difficult to locate some of the inlet points. Due to this, the majority of the holes were not used. Also, while attempting to locate unblocked injection points, the adhesive was noticed to have cured inside some of the syringe needles and on the injecting surface. Figure 8.9 shows a schematic representation of the inlets at the blade’s root. Furthermore, outflow of the cyanoacrylate adhesive was noted at the blade’s tip. Traces of the outflow path was observed on the blade from the tip to the mid section where the blade was being loaded, Figure 8.10.

Figure 8.9: Schematic illustration of the injecting point at the blade’s root

Close to concluding the ‘at failure’ test run, there was a sudden increase in load due to the maximum deflected part of the blade, at the loading point making contact with the steel base on which the wooden side supports were placed. This led to a 15 mm cut on the underside of blade near the loading timber, Figure 8.11a. The cut was at the leading edge and also led to a significant bending which tilted up the blade’s tip. Outflow of the adhesive was also noted at this damaged site.

Post-repair

Figure 8.12 shows the NACA 4415 blade’s post-repair bending behaviour along with those from the pre-repair stage. This figure clearly demonstrates the influ-
8.5 Results and Discussion

Figure 8.10: Loctite 406 flow path from the blade’s tip

(a) Cross-sectional view of the blade’s root  (b) Enlarge view of the of the curvatures

Figure 8.11: Underside cut on NACA 4415 and the tilting effect
ence of the CA adhesive in aiding considerable recovery of the blade’s stiffness. The flexural stiffness of the repaired blade, within the elastic region between 100-700 N, revealed a stiffness recovery of approximately 72 Nm². This stands at approximately 84% of the initial flexural stiffness. The recovered efficiency and the comparison plots in Figure 8.12 indicate that the adhesive was solely responsible for the linear portion of the ‘post-repair’ plot. Beyond the elastic region, the adhesive’s bond strength dwindles as the blade gradually reverted to its normal non-linear damaged behaviour.

![Figure 8.12: Post-repair and pre-repair bending test results on NACA 4415](image)

The post-repair failure was of similar nature to the pre-repair one but the underside cut experienced further damage. Therefore, it can be said that successful adhesive injection from the first row, closer to the loading plane, was instrumental...
8.5 Results and Discussion

Figure 8.13: Gradients of the results using secant modulus

in repairing some of the delamination, though it was not quantified by area, but the adhesive was observed flowing into this area. This in effect contributed to the overall recovery of the blade.

The results provide validation of the concept that complex structures can accommodate a self healing system. The NACA 4415 WTB was the first of its kind to be fabricated with an embedded self healing system and therefore it was expected to possess some flaws. A cross-section at the blade's root revealed an imbalanced resin concentration across the width and depth of the section, especially after each placement of the nylon cables, Figure 8.14. Also, the figure shows curvatures around the nylon cables. These curvatures can be attributed as one of the factors that led to the delamination on the blade around the loading area. Under this current self healing technique, curvature around the hollow sections
will be inevitable but should be minimised. Generally, the fabrication process can be improved, using a prefabricated casing that allows the hollow-preforming materials in the transverse direction and those along the edges to be placed at appropriate angles.

Figure 8.14: Curvature and imbalance resin volume across the width and depth
Chapter 9

Conclusions and Recommendations

9.1 Summary and Conclusions

The objective of this thesis was to devise a mechanism by which a GFRP composite structure can perform an intrinsic self repair function. This would mimic vascular system in natural organisms and with minimum infringement on the path of the healing agent. It goes further into implementing the technique into a fully functional composite structure. Also, the degree to which cyanoacrylate (CA) adhesives can be effective as a self healing agent in combating delamination at different environmental conditions was also investigated.

The research commenced through some preliminary inquiries into some of the causes behind failures of GFRP structures with special focus on wind turbine blades (WTB). The test carried out in Chapter 4 assumed that the failure of WTBs, caused by defects such as delamination, occurs naturally due to out of plane stresses at certain segments on the blade. The identification of such stressed zones was experimentally investigated on a 6 kW glass fibre-polypropylene WTB and the finite element model was also developed. Both the static and the dynamic tests conducted on the blade showed that the highest level of recorded strains emanated from the strain gauge at 20% of the blade’s length. This is contrary to what a simple beam theory would have predicted. Similarly, the finite element
model—using Tsai-Wu failure criterion—validates the point that the likelihood of the blade failure, from accumulated operational stress, will stem from the region within the third length of the blade from the root. This exception to the beam theory is attributed to the blade's asymmetric profile. This finding shows that medium size WTBs exhibit similar trace as the mega scale ones on the vulnerability sections along the blade's length. These results can be viewed as crucial for strategic positioning of a self healing system when it is incorporated into a structure like WTB.

In Chapter 5, the fracture toughness of a CA adhesive was quantified from the perspective of different environmental conditions through a double cantilever beam (DCB) test. The test showed that there exists a certainty that a self repaired section of a structure would have sufficient reinforcement against out of plane stresses thereby averting delamination. Encouraging results were discussed for the two fracture toughness experiments in the chapter on two phases — woven glass fibre composite under ambient condition and the simulated environmental impact using unidirectional glass fibre composites. At the repair stage for the woven fibre composite specimens, the average fracture toughness from DCB test for the pristine group of specimens was 337.95 N/m rising to 424 N/m upon re-bonding of the delaminated halves, Table 5.3. This represents 126% of toughness recovery. Furthermore, the adhesive was instrumental in aiding the material against interlaminar fracture as the strain energy released during the test increased by 15% from 1354 J for pre-repair to 1563 J for post-repair. The standard deviation involved in the aforementioned strain energy at both stages of repair reduced from 210 J to 79 J upon repair. Such reduction is attributed to the rubber additive component of the adhesive and the possibility of the adhesive's age given that the loads involved at both stages were quite identical. The fractography examination of the pre-repair adherends' surfaces reveals a matrix bond dominated failure—polyester matrix peeling off from the adjacent surface. However, the post-repair results show that the adhesive formed a strong bond with the exposed glass surfaces and the polyester matrix. Failure was at the interface between the adhesive and the matrix.
9.1 Summary and Conclusions

In the test involving simulated environmental conditions, coupons repaired with CA adhesive Loctite 435 displayed impressive fracture loads and initial toughness values, demonstrating the effectiveness at repairing delaminated GFRP material. Furthermore, the CA repair appeared to be durable under extreme environmental conditions. The DCB tests also demonstrated the superior propagating crack arrest abilities of the Loctite 435 adhesive to the initial polyester matrix bond.

The average fracture toughness for all the specimens increased from a value of approximately 183 N/m at pre-repair stage to a value of 265 N/m at post-repair. This represents a fracture toughness gain of 45%. The most successful repair was observed for the specimens subject to a 5% salt spray environment at 35 °C. The average fracture toughness for these specimens increased from 203 N/m for the pre-repair condition to a value of 409 N/m for the post-repair cases. This represents a toughness gain of approximately 100%.

The possibility that the entire adherend's surface was insufficiently coated by the repairing agent provides a scope for further improvement in the method of application and delivery of the agent. It was observed that specimens having the CA adhesive in their mid-plane and exposed to -20 °C temperature had slight loss of their resistance to delamination whereas those exposed to simulated corrosive environmental condition of different level of salt concentration (0% and 5%) appeared to have benefited from such exposure. Although, the Loctite 435 bonded specimens were more brittle than the initial polyester bond, the crack initiation loads were observed to be almost unchanged for the repaired specimens. Some significant increase were recorded for the case of those exposed to the saline environment. The brittleness and the stick-slip behaviour that characterised the repaired coupons are attributed to improper register of the adherends during repair.

Chapter 6 addressed the prime focus of this thesis. The first major test described in the chapter showed the conceptualisation of a 2D vascular self healing system,
9. CONCLUSIONS AND RECOMMENDATIONS

without a foreign hollow wall. The technique was proven effective in discharging repairing fluid, Loctite 435 into damaged areas. This approach was subsequently advanced and transferred onto a more intricate and complex specimen. The test reported in the first phase (dealing with bulk sized laminate), was aimed at employing a bio-mimetic and hydraulic aided approach to further enhance the self healing capabilities of FRP composites. The technique bypassed the use of hollow glass-fibres (HGF) as a delivery passage for repairing agent to a damage site, and adopted the use of single part adhesive as the healing chemical contrary to the widely used two-part compounds.

This procedure was justified in that, since HGFs were absent, no additional fracture energy is required in penetrating the HGFs' walls before repairing chemical is released onto the crack path. This removes the intermediary between the repairing agent and the propagating crack, while also eliminating the probability of non-mixing between the adhesive and its hardener or catalyst in the case of two-part repairing chemicals. The three-point flexural bending test results showed the average mechanical stiffness recoveries of 86% and 81% for the drilled and undrilled stages respectively. These represent substantial fractions of their capacity at their undamaged state. The closeness of these figures indicates that no severe structural detriment was caused by the drilling action. At these efficiencies, the structural integrity can be maintained while also averting serious structural disintegration.

The second phase demonstrated the transfer of the 2D delivery system into smaller and slender FRP laminates. This was advantageous in the light of avoiding induced damages arising from artificial incorporation of the hollow network into a fully cured laminate through a drilling process. This meant that less compromise on the material stiffness through the inclusion of the hollow network. The 3 mm hollow diameter represents 7% of the cross-sectional area of the specimens from the first phase. However, the hollow diameter was reduced below the macro-meter range for the second phase. The hollow diameter of 400 μm used represents 0.17% of the cross-sectional area of the new specimen (the slender ones). The specimens
from the group subjected to flexural damage test using a three point bending test, showed encouraging results that justified the delivery method. The results show that delaminated material or structure can be returned to almost its pristine stiffness abates some mechanical loading capacity. The specimen with delamination failure recovered 97% of its stiffness and over 90% of its loading capacity.

The introduction of the adhesive caused a change in the mode of failure from delamination (pre-repair) to flexural failure (post-repair). The predominant failure mode was the flexural failure, which was the failure mode for the remaining specimen in the group. An average of approximately 85% flexural stiffness was recovered for the remaining specimens and corresponding load capacity of 46% was also recovered. The low recovered loading strength is due to the following two reasons:

The first is due to a limited bond strength of the adhesive, resulting in a maximum of 60% of the loading capacity. This downgrades the impressive results of the recovered stiffness. Also, the primary focus was plying on the adhesive’s viscosity to evaluate the effectiveness of the hollow network (of micrometer range channels) in favouring the circulation of healing fluids. To a large extent, this was achieved considering that lowering the adhesive’s viscosity compromises or reduces its bonding strength.

The second reason may be attributed to fracturing of the glass fibres from flexural damages. Upon repair, broken fibres are not bonded back together efficiently. This signifies that when long fibres are broken down they support less load. This is the premise of almost impeccable mechanical responses of LD1 in strength and stiffness.

The fatigue tests carried out revealed some lapses in the delivery method. The test showed that potential delivery system must be within reachable proximity of likelihood of failure. Such precise positioning will enable micro damages in form of matrix cracking, fibre fracture and other forms of fatigue damage to be easily addressed. In some cases, there was no apparent recovery. The stiffness reflected the traces of declining strength, which was associated with infringements on the
healing agent’s path to the damage areas below the mid-plane, as well as the applied load at post-repair testing exceeding the adhesive’s capability. While the former claim cannot be substantiated as there was no post-repair non-destructive test (NDT) to internally examine the samples, the latter claim was validated by the results from the diminishing loading capacity group. Also, it was discovered that pressure differences at the adhesive injection point and inside the specimens led to low volume of adhesive being injected and consequently, low strength recovery. This shows that a functioning pressure-assisted self healing delivery system would dispatch its healing agent through the least pressure resistance path. Damage paths directly or indirectly connected to the delivery plane would create the low pressure areas necessary to attract the repairing fluid onto the damaged path.

The test conducted in Chapter 6 showed a potential shortcoming of the healing system. The system could be made more efficient by locating the delivery system at the most accessible stacking position. In that case, the growth of local stress concentration elements can be combated early on at the initiation stage. This issue was numerically investigated and explored in Chapter 7. An approach of stiffness reduction coefficient (SRC) in the form of a degrading model, MPDM, was applied to the laminate for failure detection. It enabled the unveiling of the most fragile and vulnerable layer(s) in the process.

Numerically simulated-three point bending test, on a cross-ply laminate showed as expected the outermost layers to be the most susceptible layers to flexural damage by tension at the bottom layer and compression at the top. The results revealed the layers with transversely oriented fibres, especially those further from the mid-plane to be the most sensitive layers to failure. In addition, the model was compared with an experimental result. The model appeared to have similar load deflection profile within the elastic range. The flexural stiffness of 205 Nm² and 197 Nm² for the numerical model and the experimental test respectively, further reinforces the similarities in the results. This, in effect suggests that the self healing system positioning in a FRP structure can be performed within reasonable accuracy. Also, the corresponding load and deflection values at which the
9.2 Key Findings

The concluding experimental test was carried out on a 500 W NACA 4415 WTB. The WTB was in-house-made with 15 layers of glass/polyester and was laced with 450 \(\mu\)m (diameter) 2D hollow networks at 2nd, 6th, 9th and 13th layer. The hollow network, a representation of a vascular system, at each of its position had a 0.025 m\(^2\) coverage of the blade’s surface area centred at a third of the blade’s length from the root. This represents 70% area of the WTB’s third length from the root. Under flexural bending test, delamination was observed near the loading point. The cause was attributed to stress concentration due the applied load. No flexural failure was observed or noticed. Rather the blade was observed to have experienced a change in behaviour from linear elastic to non-linear. This was confirmed upon reloading, and it was evident as the flexural stiffness was reduced from 87 Nm\(^2\) to 55 Nm\(^2\). This represents a 35% reduction in flexural stiffness. However, 84% of the initial stiffness was recovered upon infusing some of the inlet channels of the network with a small volume of Loctite 406. The mechanical properties after the repair were largely similar to those of the initial state within the elastic region. Beyond the elastic range, the blade simply reverted to its damaged state response. This clearly shows that the post-repair material mechanical function will be comprehensively dependent on the repairing agent. Also, the hollow section was effective in delivering the adhesive to the delaminated site. The CA repairing fluid was observed flowing towards the delaminated area.

9.2 Key Findings

The key findings from this thesis are outlined in the succeeding paragraphs.

It was observed that complex thin-walled structures, FRP WTBs in particular, have the propensity to develop local stress concentration around one third of the length from the root. This appears to be common feature especially for both small and large scale WTBs, which are of complex aerodynamics shape profiles.
This thesis showed that out-of-plane stresses in FRP structures arising from either internally generated local stress concentration or those initiated by microcrack formation, can be combated through polymer toughening. The initial polymer in-between the affected FRP layers was coated with a tougher compound, such as CA adhesives. In mode I DCB delamination test, cyanoacrylate adhesive was robust in addressing interlaminar fracture under varying degrees of different stringent environmental conditions, such as when exposed to saline and extreme temperatures. It was found that on average, under the stated conditions, the fracture toughness, $G_{ic}$ can be improved by 48% using a CA adhesive.

The proposed vascular system for the healing agent's transportation in a self healing system was successfully designed and implemented. The 2D hollow network channels were first incorporated into small GFRP coupons and then, into a GFRP NACA 4415 WTB. The hollow channels were discovered to be effective in carrying out autonomous repair and efficient due to their small volume. The healing technique ensured an average of 84% and 46% for flexural stiffness and mechanical loading capacity recoveries respectively. These operational efficiency were obtained for GFRP coupons whose hollow network was at the mid-plane, a relatively safe distance from the flexural hotspots.

The NACA 4415 WTB was manufactured with four layers of the 2D hollow network within its fifteen layers glass fibre polymer matrix. Three-point bending test on the blade produced some delaminated areas and a change in the mechanical properties. The loading produced a non-linear deformation in the blade. However, when the healing system was used with Loctite 406, 84% of the mechanical properties in term of the elastic flexural stiffness was recovered. Also, reduction in the delaminated area was observed.

A proposed method for PPFA showed the numerical model and experimental results to be of similar nature. The flexural stiffness of 205 Nm$^2$ and 197 Nm$^2$ for the numerical and the experimental respectively are within a close range of each other. The model does not accurately predict the experimental behaviours due
9.3 Recommendations

The 2D vascular network for adhesive delivery was largely successful. However, it will be recalled from the preceding chapter, Chapter 8 that there were resin infested curvatures above the injection points, Figure 8.14. Such high concentration of resin is detrimental. It would be interesting to device a specialised system of fabricating self healing material or coupons to minimise the curvatures. This will involve using smaller diameter of hollow preform material, similar to the size of the fibres. Also, a comprehensive finite element analysis of the curvature effects on the neighbouring fibre and the whole laminate would advance the knowledge of the automated healing system.

The use of commingled fibres holds a prospective avenue in reducing or eliminating resin pockets. This will involve the intended reinforcing fibre clothing being laced with fugitive material (e.g micro-crystalline wax) of similar thickness to the fibre strands, at the appropriate interval, where the vasculatures will be located. In simple terms, it is the construction of the primary (either glass or carbon) fibre mat, around a configured vasculature material; a fibre strand in the mat being replaced by a transitory or ephemeral substance. Such 'semi-hybrid' fibre clothing can be used normally in fabrication of hollow network FRP composites. The post-curing processing of the composite will include the extraction of the fugitive material either chemically or through thermal treatment of the composite at the appropriate temperature.

Another interesting aspect of self healing is the automation of the repairing system (ARS). Damaged site needs to attract the repairing fluid. Therefore, an
9. CONCLUSIONS AND RECOMMENDATIONS

An automated pressurised system of delivering the healing agent would be essential. A possible avenue of transcending the ARS developed in Chapter 6 would be to embed strain sensors within the layers of a FRP laminate. A further developed ARS introduced in this thesis, used in conjunction with fine-tuned PPFA, would be an important asset to self healing. The use of commercially available software packages for PPFA will present an advancement and improvement in the ability to predict failure—its type, location, and size—for different structural loading conditions and with array of different failure criteria.

The vascular system developed in this thesis is rather an implementation and cannot at all be considered a fully efficient system. An optimisation of the healing network would be a step forward. The delivery network should be efficient and sustainable from the viewpoint of minimising; the mechanical power for the healing agent's circulation, adequate network size and hollow diameter etc. Also, the system's life span and optimum number of repair cycles should be considered.
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Appendix A

Data Reduction Method for Mode I Fracture Toughness, $G_{IC}$

A.1 Introduction

In the course of running an experiment on either of the first two delamination modes, quite significant amount of data can be generated. There are numbers of available schemes to reduce the accumulated data for close representation of experimental observations. First, the DCB is originating from simple beam theory and the majority of the reduction methods are based on linear elastic fracture mechanics (LEFM). It is assumed that the material already contained a crack to which when loaded causes increased crack width or crack area thereby changing the energy release rate $G$, also referred to as the fracture toughness i.e. the rate at which the strain energy, $U_s$ changes with respect to surface area, $A$. The strain energy can be written as:

$$U_s = \frac{1}{2} P \delta \quad (A.1)$$

and the fracture toughness as

$$G = \frac{dU_s}{dA} \quad (A.2)$$

given that $dA = B da$ where $B$ is the thickness and $a$ the crack length. Using
equation A.1 and equation A.2, change in strain energy, dU can be written as

\[ U_s = \frac{1}{2} (P \delta + \delta P) \]  

(A.3)

hence,

\[ G = \frac{1}{2b} \left[ \frac{P \delta}{da} - \frac{\delta P}{da} \right] \]  

(A.4)

Equation A.4 can be reduced to a compact form by engaging the compliance, C. where

\[ C = \frac{\delta}{P} \quad \implies \quad \delta = CP \]

taking the derivative and followed by appropriate substitution

\[ d\delta = CdP + PdC \]  

(A.5)

\[ G = \frac{P^2}{2b} \frac{dC}{da} \]  

(A.6)

or

\[ G = \frac{P\delta}{2b} \frac{1}{C} \frac{dC}{da} \]  

(A.7)

A.2 Corrected beam method (CBT)

The CBT represents the modified version of the simple beam theory noting the absence of perfectly built-in support for the DCB specimens, which might otherwise underestimate the compliance. Hence, the CBT assumes longer crack length, \((a + |\Delta|)\) than the effective crack length, \(a\) on the specimen. \(|\Delta|\) is the additional crack length which is obtained from cube root plot, Figure A.1, of the compliance
A.3 Area method

against the delamination or crack length, \( a \) i.e. \( C^\frac{1}{3} \) vs \( a \). \(|\Delta|\) takes the magnitude of the x-axis intersection of the best linear plot. The \( G_{fc} \) is given by:

\[
G_{fc} = \frac{3P\delta}{2b(a + \Delta)}
\]  

(A.8)

where \( P \) is the load, \( \delta \) the displacement, \( a \) the crack or delamination length, and \( b \) the specimen’s width.

Figure A.1: Obtaining the correction factor, \( \Delta \)

A.3 Area method

In this case, the reduction method works on the principle that for linear elastic body strain-energy used in crack extension, \( da \) is equal to the area between the loading \((\delta_i, P_i)\), and unloading points \((\delta_{i+1}, P_{i+1})\), Figure A.2. This is based on the assumption that both \( da \) and \( dP \) are not finite and can be approximated as
A. DATA REDUCTION METHOD FOR MODE I FRACTURE TOUGHNESS, $G_{IC}$

A straight line (Bardis & Kedward, 2004). The fracture toughness is calculated directly by making slight changes to the rate of change of strain-energy in equation A.4. The $G_{IC}$ is obtained by taking the average strain-energy of other subsequent loading and unloading points as:

$$G_{IC} = \frac{1}{2b} \sum_{i=1}^{n} \left[ P_{i} \delta_{i} - \delta_{i} dP_{i} \right]$$  (A.9)

Figure A.2: Area method’s load-displacement plot, after Szekrnyes (2005)

A.4 Compliance Method

The interlaminar fracture toughness can be computed from this method by obtaining the derivative part of equation A.6 from the plot of the compliance $C$, against the delamination length, $a$ and substituting the value back into the equation. This method is rarely used due to irregular, serrated plots, otherwise known as stick – slip and the associated difficulty in fitting them.
A.5 Simple Beam Theory

In some experimental cases the unstable crack propagation renders inappropriate the other reduction method already outlined. Following equation A.6, $\frac{dC}{da}$ can be expressed as:

$$\frac{dC}{da} = \frac{8}{E_s b} \left( \frac{3a^2}{h^3} + \frac{1}{h} \right)$$

(A.10)

Substitution equation A.10 into equation A.6 gives

$$G_{ic} = \frac{4P^2}{E_s b^2} \left( \frac{3a^2}{h^3} + \frac{1}{h} \right)$$

(A.11)

where $h$ is the half thickness of DCB specimen, and $E_s$ is the independently measured tensile modulus of one half of the substrate. This approach sidesteps the dependency on the load deflection curve. However, the need for the elastic modulus is a drawback as this property tends to vary between specimens and may not always be available.
A. DATA REDUCTION METHOD FOR MODE I FRACTURE TOUGHNESS, $G_{IC}$
Appendix B

Chapter 5: Load-Displacement plots

Figure B.1: DB-2: Load-Displacement
B. CHAPTER 5: LOAD-DISPLACEMENT PLOTS

Figure B.2: R-Curve: Fracture resistant of DB-2

Figure B.3: DB-5: Load-Displacement
Figure B.4: R-Curve: Fracture resistant of DB-5

Figure B.5: DB-6: Load-Displacement
Figure B.6: R-Curve: Fracture resistant of DB-6